VACUUM

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FREEZE DRYING SYMPOSIUM

DRYING by vacuum sublimation or freeze drying as the technique is now so commonly called, continues to make its spectacular contribution to medical and veterinary work, closely followed by promising applications in food processing. Documentation of these developments is scattered throughout the current literature and there are a few books available on this subject, notably Flosdorf's ' Freeze Drying,' but books are soon overtaken by the speed of events although valuably serving as mile stones of progress.

It is significant that the newly formed Institute of Biology decided to dedicate a major organised event in its young history to a symposium of freeze drying. The conference took place in June last and the review published in this issue clearly shows its success. Not only was it proved that vacuum sublimation is of profound interest to a large number of practising scientists—some 400 attended—but the conference rendered a valuable service by bringing up to date our knowledge of freeze drying as applied in medicine and associated fields.

VACUUM AS A PIONEERING PROCESS

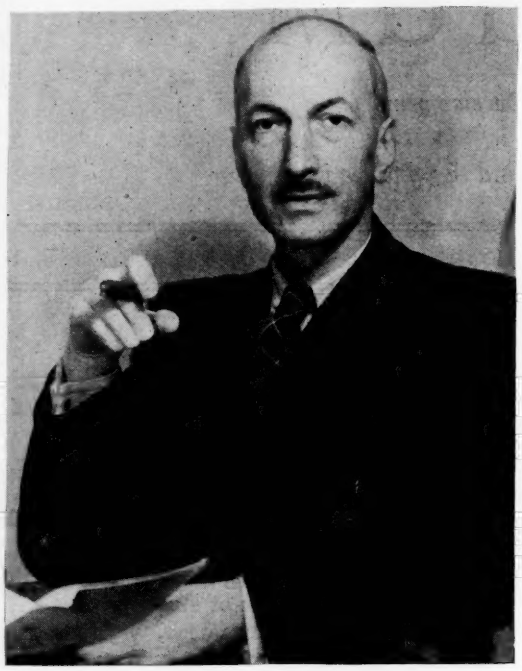
WE HAVE NOW COME to expect and plan that vacuum practices initiated in the laboratory, will become full scale manufacturing operations, either for the provision of a vacuum in the finished article, or for processing *in vacuo*. Many important processes carried out in a vacuum have established themselves in the face of strong competition from existing techniques, either because they produce a better (and sometimes even a cheaper) article, or one not hitherto realisable.

It may not be generally appreciated, however, that vacuum processing has also a potentially powerful part to play as a pioneering process. It can be of supreme help in concluding the first—often the most difficult—step of producing sufficient initial quantities for large-scale evaluation of a new product. When the product has proved itself, vigorous method research can lead to non-vacuum production procedures.

Take penicillin for example which was discovered in the course of bacteriological investigations. In attempts to isolate active material it was found that penicillin in solution was unstable. This difficulty was eventually overcome by applying freeze drying techniques, and penicillin was made available in sufficient quantities, unequivocally to prove its value. Persistent research work into production procedures yielded the new methods, now in use, of stabilising penicillin by means of crystallisation at ordinary pressures.

This is a typical case of vacuum as a pioneering process, its role proving transitory but still essential to the discovery and establishment of a material now vital to mankind. We have no doubt that many new products will follow a similar course.

This potentiality of vacuum should be kept in mind by those engaged in the discovery or development of new materials or processes, for it may provide in a relatively easy manner, the first essential step for the bulk production of an important new product, from which the normal atmospheric process can be evolved.



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Dr. von Engel was born in Vienna in 1898. He studied at the Technische Hochschule, Darmstadt (Germany), where he graduated in 1922. He continued studying natural sciences and was awarded a doctorate in 1929.

Dr. von Engel concentrated on research work very early in his career. During the years 1922-1937 he was engaged in work at the laboratories of the Siemens concern in Berlin, specialising in the investigation of high frequency problems and electric discharge phenomena in gases. During that period he became a member of the German Committee of Units and Formulae and was finally appointed Chairman of the Sub-Committee on Electric Discharges. In the years 1932 to 1934 he wrote, jointly with Dr. M. Steenbeck, a text book on Electric Discharges in Gases, which became very well known and the demand for which was so great that it had to be reprinted in the U.S.A. during the war.

In 1940 Dr. von Engel came to this country. He joined first the Electrical Laboratory at Oxford and later the Clarendon Laboratory under Lord Cherwell, resuming his research work on ferro-magnetism and on electric phenomena in gases. He is still engaged in these studies, leading a group of post-graduate students and, in addition, acts as a tutor in physics.

Dr. von Engel is a member of Christ Church and a Lecturer in Physics at Exeter College, Oxford. He became an M.A. in 1943 and took the degree of D.Sc. in 1946. A year later he was awarded an I.C.I. Fellowship and this year he was called to the U.S.A. as Visiting Professor of Physics at the University of California, Berkeley. He received a Fulbright grant which enabled him to tour the U.S.A. extensively and to visit many Universities and Research Laboratories in that country.

NEW TRENDS in vacuum research based on IONISATION PHENOMENA

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Summary

THE RANGE OF pressure measurement of the McLeod gauge and the Philips ionisation gauge has been extended recently. For the measurement of ultra-high vacua only the thermionic ionisation gauge can be used, but in such a form that the effect of soft X-rays is reduced considerably. The ionisation gauge also acts as an ionisation pump giving vacua of about 10^{-11}mm. Hg. A method of producing very pure helium gas from liquid helium II is described.

Sommaire

LA GAMME de mesures de pression de la jauge McLeod et la jauge à ionisation Philips à récemment été agrandie. Pour la mesure des vides très poussés, seule la jauge à ionisation thermionique peut être utilisée, mais de sorte que l'effet de rayons X mous soit considérablement réduit. La jauge à ionisation agit en pompe à ionisation, donnant des vides d'à peu près 10^{-11}mm. Hg. L'on décrit une méthode de production de gaz hélium très pur partant de l'hélium II liquide.

INTRODUCTION

ABOUT TEN YEARS AGO it appeared as if high vacuum technique was approaching its ultimate state of development. Then the lowest pressure which could be obtained in exhausted vessels either permanently connected with the pumping system or sealed off after pumping was considered to be of the order of 10^{-6}–10^{-8}mm. Hg. The production of low pressures was not the only problem however ; in addition, the time required to produce it, had to be reasonably small. From the commercial point of view this factor is important, as large numbers of vessels have to be evacuated quickly whilst, in the scientific field, large volumes have to be kept continuously at a low pressure.

During a visit to the United States this year, I learned that by application of ionisation processes high vacuum technique has recently taken a new turn. Though the specialist in this field may be well acquainted with these facts, I propose to discuss in this paper some of these new ideas for the benefit of the 'general physicist' and try to indicate finally the way in which these may reflect upon physical research.

Closely connected with the problems concerning high vacua are the problems related to the purification of atomic and molecular gases. I shall briefly outline certain advances which have been made in this field, too.

One might think that any new developments regarding the production of high vacua would result from improvements made to existing pumping methods and that progress in measuring such high vacua would follow later. Here the reverse is the case. A completely new pumping method originated from research into the method of measuring. Consequently we shall deal with the methods of measuring first, and the production of high vacua later. The history of the apparatus required for producing high vacua has been dealt with by Professor E. N. da C. Andrade in the first issue of this journal.

*M.S. received October, 1951

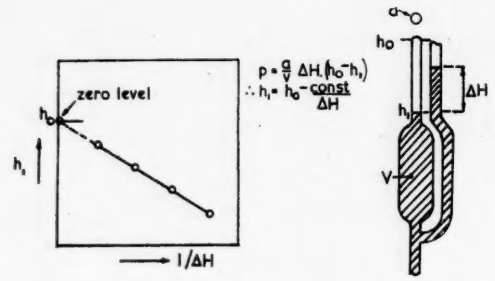

Fig. 1. The McLeod gauge. An accurate value of the pressure is obtained, when the zero level h_0, is taken from the extrapolated curve $h_1 = f(1/\Delta H)$.

MEASUREMENT OF HIGH VACUA

Let us first look at the limitations imposed on the conventional methods of measuring low pressures[1,2]. The McLeod gauge is still used as an absolute manometer in all those cases where the pressure does not fall below 10^{-6}mm. Hg. But the accuracy of measurements obtained with this type of gauge is often very poor at the lower end of the pressure range and there is a limit to the speed at which single measurements can be taken. Thus, the McLeod gauge cannot serve as a recording device, should pressure changes occur during a run; furthermore, this gauge does not measure the pressure of condensable vapours. Finally, it does not give an indication regarding the composition of the gas mixture or the nature of the gas.

Recently a number of suggestions have been made to improve the McLeod gauge and to obtain higher accuracy of measurement at low pressures. For example, instead of measuring the difference in height of the mercury levels, $\triangle H$, in the capillary tube containing the compressed gas sample and the capillary tube connected to the evacuated vessel, a series of readings of the heights of the mercury levels are taken by telescope for various positions of the level h_1 in the left tube (Fig. 1). From the graph showing $h_1 = f(\triangle H)$ the pressure can be derived without reference to a fixed zero level, h_0, which is usually taken to be the upper end of the capillary tube. Applying this method one can avoid serious errors which may be introduced through lack of proper definition of the zero level. It is well known that the inner surface of the sealed end of a capillary tube is hemispherical in shape rather than plane and since the measurement of very low pressures makes it necessary to determine differences of height equal to 1 mm. or less, the readings obtained in that range may indicate the order of magnitude of the pressure but nothing more.

Another way of increasing the sensitivity of the gauge is to compress several samples of the gas in the capillary tube by means of a valve thus increasing the difference in the height of the two levels.

Certain difficulties are encountered when the pressure of vapours is to be determined[3]. By means of a Knudsen-leaf gauge vapour pressures down to about 10^{-6}mm. Hg have been measured[4].

An instrument used for the measurement of pressures down to lower values than those covered by the McLeod gauge is the Philips gauge. Here the current flowing through the evacuated space in the form of a discharge is a measure of the gas pressure. The gauge consists essentially of a vessel in which a hollow cathode glow discharge is maintained in the presence of a magnetic field (Fig. 2). Its working principle[5] is as follows : if a potential difference of several thousand volts is applied between two cold metal electrodes in a gas a glow discharge will develop. A steady flow of current can be established only if each electron which is emitted by the cathode produces a number of positive ions and electrons on its way to the anode and if these ions (together with the metastables and light quanta produced by the discharge) will liberate just one further electron on striking the cathode. This process can continue only, while the potential difference between cathode and anode is sufficiently high. If the pressure is reduced to 10^{-3}mm. Hg or less, the potential difference required rises very rapidly because the initial electron finds fewer molecules along its path, with which it can collide, and thus the energy of the electron is transferred to the anode instead of to the gas. By applying a magnetic field – those produced by permanent magnets have proved to be sufficiently strong for the

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purpose – the electron, instead of travelling in the direction of the electric field, acquires a component of velocity perpendicular to both, the magnetic and the electric field. Consequently it spirals along the magnetic field lines ; its path is thereby lengthened and it collides more often with gas molecules on its way to the anode. It can be shown that the comparative increase in the number of collisions is roughly proportional to H/p^2, where H is the magnetic field (usually 300 – 8,000 0e) and p the gas pressure[6]. It will be realised that the provision of a magnetic field has the same effect as if the pressure of the gas was increased by a large factor. As a result a discharge can now be maintained with the same potential difference as before although the pressure has been reduced.

The effect can be improved still further, i.e. the current can be greatly increased, if the single cathode is replaced by a pair of cathodes facing each other (Fig. 2) – an arrangement called a ' hollow ' cathode – and, if the cathode surfaces consist of zirconium or thorium which show a large secondary electron emission. It is interesting to note that, although this type of gauge has now been known for some time, no satisfactory treatment of its discharge phenomena exists.

However, the conditions for the starting of a low pressure gas discharge in the presence of a magnetic field have been recently investigated both theoretically and experimentally by G. Valle[7]. The Philips gauges can now be applied in a pressure region down to 10^{-7}mm. Hg. At still lower pressures the large voltages required would make the use of this gauge impracticable. Incidentally, there is one aspect of its operation which makes this type of gauge a useful tool for determining the nature of the residual gas in exhausted vessels : the radiation produced in the discharge gives a spectrum which can be easily identified (provided that the electrodes are sufficiently clean) and this effect helps to locate a possible leak or source from which the residual gas is evolved. Also, by using small mass spectrometers the nature of the residual gases can be analysed, a method which is being applied in leak detection.

A different method of extending the measuring

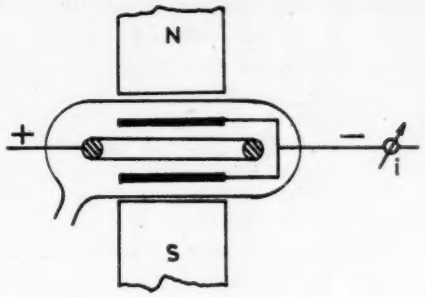

Fig. 2. Diagrammatic view of the Philips gauge.

range towards still lower pressures is available in the form of the thermionic ionisation gauge[1]. Here the pressure is determined by the magnitude of the current which is caused by the ionisation of gas molecules by electrons. An ordinary hot cathode ionisation gauge (Fig. 3) has, for example, a straight filament mounted along the axis of a wire mesh anode, a third electrode, the collector, surrounding the anode coaxially. If a potential of 100 V or more is applied to the anode, the cathode being at zero potential, electrons will be emitted from the cathode which, having acquired sufficient energy, will ionise gas molecules. Some of the positive ions will return to the cathode, partly neutralising the negative space charge and partly being neutralised at the cathode surface, whereas the major portion of the electrons will flow to the anode. However, those electrons which pass between the wires of the anode with sufficient speed will enter the collector space and produce in it ionisation by collision. Since the collector is kept at a potential of the order of 10 V negative with respect to the cathode, positive ions formed will be attracted and electrons will be repelled by the collector. Since the number of ionising collisions is proportional to the gas density, the number of ions formed, and hence the number of ions flowing to the collector, is proportional to the gas density and the electron current.

It has been known for quite a time that at very low pressures the collector current does not fall when the pressure is further reduced, and also that for a given collector potential, the collector current - anode

voltage characteristic remains practically the same. This residual current was a mystery for many years[1].

Two suggestions for the cause of the residual current have been advanced : (a) the emission of electrons by X-rays falling on the collector and (b) the emission of ions from the anode[8].

If electrons of approximately 100 volts strike the anode, soft X-rays are emitted. Unless the gas present is of a sufficient density, the X-rays pass through the space without being absorbed. Since the anode consists of a cylinder with slits, or a wire grid, X-ray radiation emitted from the inner surface of the anode (Fig. 3) can easily penetrate to the collector and release electrons. The potential of the anode is 100–300 volts positive with respect to

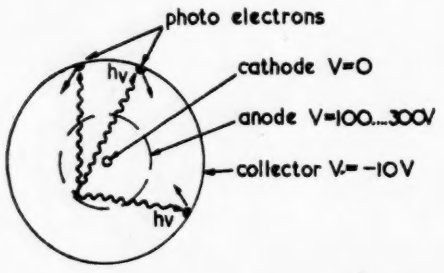

Fig. 3. Conventional electrode arrangement of an ionisation gauge. Electrons from the central filament striking the anode produce soft X-rays which release photo-electrons from the collector.

the collector. An electron current will, therefore, flow from the collector to the anode which is equivalent to a flow of ions from the gas to the collector. As a result the galvanometer in the collector circuit will indicate a current which would be present, even if no residual gas were left in the gauge.

It might be useful to give here a numerical example as an illustration of the foregoing. An average value for the observed sensitivity of an ionisation gauge operating in Argon is $\sim$ 0.01 mA collector current per mA electron current at 1 μ pressure, 250 V being applied to the anode. Let us assume that each electron, instead of moving with a speed which rises as its distance from the cathode increases, travels with a constant speed corresponding to say 100 eV. We know the value of the efficiency of ionisation caused by single collisions in Argon. It is found that one electron produces about 10 pairs of ions per cm. of path at 1 mm. Hg (this figure is true for electron energies between 80 and 200 eV). Assuming an ionising path of the order of 1 cm. and a pressure of 1 μ we obtain about 10^{-2} ions per electron or an ion current of 10^{-5} A for 1 mA electron current. This result agrees with the above observations. The ion current is found to be smaller if the collector is arranged between filament and anode, because a part of the space between cathode and collector is nearly field-free and thus the section of the path, along which the electrons have high energy, is shortened.

If the gas pressure is very low, the effect of X-rays releasing electrons from the collector can become appreciable. A rough calculation will confirm this. Since the electrons arriving at the anode surface have an energy of 100 eV or more the ultimate wavelength of the associated X-rays is $\lambda = 12{,}400/100 = 124$Å or less. From classical X-ray studies the fraction of energy transformed into radiation was found empirically to be $f \sim 10^{-9} \times Z \times V$, where Z is the atomic number of the element used as anode material and V the potential through which the electrons have fallen. If $Z=50$, $V=100$ V, we have $f \sim 5 \times 10^{-6}$. Assuming that each quantum has an energy of 100 eV, that each quantum releases one electron and that the loss of radiation is zero, we find that the ratio of photo-current to electron-current equals f. Thus 1 mA emission will produce a maximum collector (electron) current of 10^{-9}A. Since the ion current was 10^{-5}A at 10^{-3}mm. Hg (see above), the ion current will be of the same order as the photo-current at a pressure of $p = 10^{-7}$mm. Hg. This again is more or less in agreement with observations.

In order to make an ionisation gauge applicable to very low pressures the effect of soft X-ray radiation has to be reduced. This can be done by using substances for the anode which have a low atomic number but it is hardly possible to reduce the intensity of radiation by more than one order of magnitude in this manner. Another possibility is the lowering of the anode potential, but this reduces the

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electron current entering the collector space at the same time, and thus diminishes the sensitivity of the gauge. A very efficient method for extending the measuring range has been used with success by Bayard and Alpert[9]. By interchanging the filament and the collector they have effectively reduced the surface area exposed to X-rays by several orders of magnitude. Fig. 4 shows the new gauge diagrammatically. Here the filament is arranged outside the anode and the collector is a thin wire stretched along the axis of the tube. Because the surface of this wire can be made more than 100 times smaller than that of the old collector, the residual current can be reduced by approximately the same factor, and thus, pressures down to 10^{-11}mm. Hg can now be measured. End plates closing the anode cylinder prevent losses of ions by axial motion.

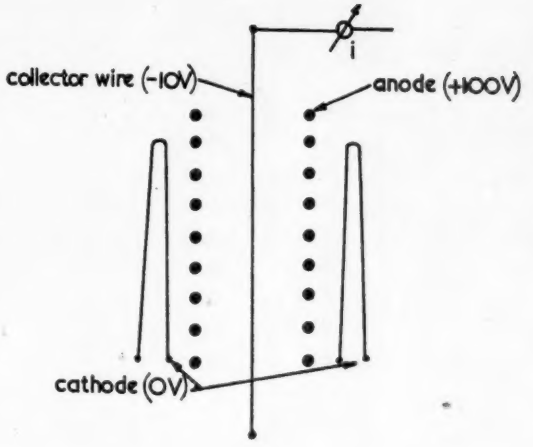

Fig. 4. New electrode arrangement in the ionisation gauge. Electrons emitted from the filament striking the anode produce X-rays. The latter can only release a few photo-electrons from the thin central collector wire. The second cathode is a spare.

CALIBRATION OF PRESSURE GAUGES FOR ULTRA HIGH VACUA

Having obtained an instrument capable of detecting very low gas pressures, the question now arises as to what method of calibration can be regarded as giving reliable results in that range. Once all secondary effects are excluded, the ionisation gauge should have an ion collector current which is strictly proportional to the gas density. If this were so, one could calibrate an ionisation gauge e.g. by means of a McLeod gauge, at say, 10^{-5}mm. Hg and then extend the calibration curve of the ionisation gauge to the low pressure region by linear extrapolation. A more elegant method is the following. It is essentially based on the measurement of the time rate of change of pressure. Two vessels are connected by a tube as shown in Fig. 5. Vessel 1 is kept at a pressure, of say, 10^{-3}mm. Hg and vessel 2 is evacuated to a very low pressure. If a tap placed between them is opened, a pressure gauge connected to vessel 2 should show a pressure which rises initially proportional to the time. The conductance C of the tube connecting the two vessels is inversely proportional to its length and directly proportional to the cube of its diameter. Its value depends, therefore, on its geometry only, provided that the gas in the system and its temperature remain unchanged. The rate of the rise of pressure p_2 of volume V_2 in vessel 2 for a given constant pressure p_1 in vessel 1 is

$$\left(\frac{dp_2}{dt}\right)=\frac{C}{V_2}p_1=\text{const.}$$

Hence $p_2 \propto t$

The constants C, V_2 and p_1 being known, dp_2/dt can be calculated. An ionisation gauge showing a constant rate of rise of collector current is thus calibrated in terms of changes in pressure, i.e.

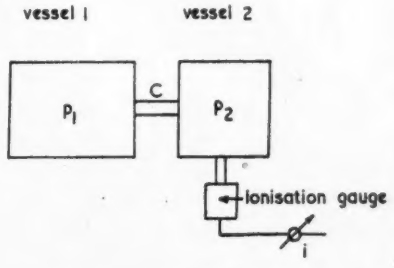

Fig. 5. Arrangement for the calibration of an ionisation gauge by the flow method based on the known value of the conductance of the tube connecting the vessels, (p_1=constant).

$\Delta i_{coll} \propto \Delta p_2$. In order to obtain absolute values of pressures an additional measurement, usually at a known higher pressure, has to be made.

PRODUCTION OF ULTRA-HIGH VACUA

The next point of interest is to show how these very low pressures can be produced. Let us assume we have an ideal pump which is capable of reducing the pressure in a vacuum system to about 10^{-11}mm. Hg. Then, we have to inquire first of all into the vapour pressures of the pumping fluids, and into the vapour pressure of the metal and glass used in the system. The vapour pressures of copper and glass at room temperature are very much lower than 10^{-11}mm. Hg. The vapour pressure of mercury at room temperature is of the order of 10^{-3}mm. Hg. Thus, liquid air traps are required if the pressure in the system is to be reduced to less than 10^{-11}mm. Hg.

However, the new vacuum pumps use fluids which have vapour pressures of between 10^{-5} to 10^{-6}mm. Hg at the temperature of the cooling water (between 8°C. and 12°C.). By means of cooled baffles still lower pressures can be attained in the system. In fact, in modern high vacuum apparatus no liquid air or dry ice is needed.

Small traces of water present in an evacuated apparatus, together with gases absorbed by the metal of the pumping lines and valves represent a most dangerous source of gas. These gases can never be completely removed. The gases dissolved in the metal diffuse continuously from the interior of the metal to the surface and hence into the vacuum. Although the amount released per unit of time may be small, there may be a still more powerful source of gas due to the leakage of certain constituents of the air through the walls of the evacuated glass vessel. Although the magnitude of this gas flow is small by our present standards, it may be sizeable by future standards once we have become more familiar with work in the region of ultra-high vacua.

Studies of residual gas phenomena of the type just described resulted in the following most important observation. Whenever a system was isolated after pumping down to the lowest obtainable pressure, an ionisation gauge connected to the system showed that the pressure in the system continued to decrease for no apparent reason. On switching off the gauge, the pressure rose again.

It has been known for quite sometime that the pressure decreases in tubes in which ionisation takes place, when an electric field is applied. These 'clean-up' phenomena have been observed in discharge tubes, in the old gas-filled X-ray tubes, in soft cathode-ray tubes, in discharge lamps used for lighting and many other similar devices. Actually, when the gas atoms are ionised, the positive ions acquire sufficiently large speeds in the electric field to be driven into the negative electrode. When positive ions impinge upon the surface of a metal electrode, they pick out at least one electron from the metal which neutralises their positive charge and a certain fraction of these atoms enters the metal of the electrode; the remainder is reflected and returns as neutral atoms into the gas. It is obvious that such a process will not depend only on the nature of the gas and the metal but also on the amount of gas already present in the metal, on its structure and on many other parameters. In order to prove that this is not a mere speculation, experiments may be quoted which Seeliger et al. carried out before the last war[10,11]. He used a glow discharge in neon at a pressure of a few mm. Hg and an iron cathode. After running the discharge for several hundred hours a considerable drop in the gas pressure was observed. This allowed him to measure the amount of gas which had disappeared. The cathode was then moved into another part of the tube, in order to prevent the release of gas from part of the walls covered with sputtered metal, and the cathode was then heated to 1,100°C. for 20 hrs. It was found that 98% of the gas which had disappeared could be recovered: it had been absorbed by the iron cathode.

As explained before, the effect of an ionisation gauge is to ionise the gas molecules and to convey the positive ions to the collector. The collector, however, does not only receive positive charges, but some of the molecules enter the collector metal[12] – in other words the ionisation gauge acts as a pump.

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The use of an ionisation pump has been advocated and its construction has often been suggested in the past, but such a device has never been tried in practice or its limitations investigated as far as I am aware. It is interesting to recall a recent experiment by Champeix[13] who used a glow discharge maintained in a narrow tube with electrodes in an adjoining chamber to reduce the pressure in the narrow part from 10^{-1} to 10^{-4}mm. Hg. In order to reduce the pressure further while keeping the discharge potential reasonably low, a magnetic field can be applied to the glow discharge.

The pumping speed of such an ion pump can easily be estimated in the following manner : let us assume that every positive ion which reaches the collector is absorbed by the metal so that the number of neutralised ions is equal to the number, Z, of gas molecules which disappear. Thus the rate of change of their respective numbers is

$$-\frac{dZ}{dt}=\frac{i_{coll}}{e} \qquad (1)$$

since i_{coll}/e is the total number of ions which reach the collector per second. The gas pressure p and the concentration N of molecules for an ideal gas obeys the relation $p=NkT$ where k is the Boltzmann constant and T the absolute temperature. Hence at a given T

$$\frac{dp}{dt}=kT\,\frac{dN}{dt} \qquad (2)$$

But $N=Z/V$ where V is the volume which is to be evacuated. Thus we have from (1) and (2)

$$\frac{dp}{dt}=kT\frac{d(Z/V)}{dt}=-\frac{kT}{Ve}\,i_{coll} \qquad (3)$$

For example at $T=300°K$ ($k=1.37\times10^{-16}$ ergs/°K), $V=10^3$ cm^3 and $e=1.6\times10^{-19}$ Coul. an ion current of 10^{-5}A is associated with a rate of change of pressure of $\sim 2.5\times10^{-3}$ dynes/cm.^{2}/sec. or $\sim 2.10^{-6}$mm. Hg per second. This is the order of magnitude of the rate of pumping which has been observed in investigations on ionisation gauges. In fact the conditions are more complex because positive ions also reach the negative filament, chemical changes and absorption of gases by the electrodes and adsorption on the glass may occur, and the effect of sputtering has to be allowed for[14]. The nature and pressure of the gas are important factors, too.

It would appear as if an increase in the ion current would increase the speed of the pump. There are, however, a number of factors which may restrict the pumping speed and, in particular, may act as a limit in obtaining still lower pressures. In the first instance we have to consider the back pressure which is set up in the interior of the metal as a result of an increased concentration of gas in the metal. This will cause a diffusion of gas from the metal into the vacuum. This effect will be enhanced at larger current densities by the heating of the metal and this can be quite considerable, since an amount of energy equal to that of the ionisation energy is liberated in the metal with each neutralisation process.

In order to achieve and maintain ultra-high vacua, it is necessary to dispense with the conventional method of using greased taps, mercury cut-offs, glass tubes etc. Metal tubing systems and metal taps which can be baked out completely have to be used. Metal-to-glass seals, in which the exposed glass surfaces are of small area, are permissible. A new all-metal tap is now used[15] which consists of a valve with a conical opening. The moveable part is a 60° Kovar cone which is pressed into a copper seat by means of a large lever which produces a force of several tons. It is claimed that the conductance of such a valve when closed is of the order of 10^{-11} litres/second, which is sufficiently low when the rise in pressure is of the order of 10^{-12}mm. Hg. per sec. as obtained in a system of 1 litre volume. The mounting of these valves is such that the large forces which are necessary to close the valve safely can be applied without transferring the stress to the metal-to-glass seal.

THE PREPARATION OF EXTREMELY PURE HELIUM

A problem which is closely allied with the production of ultra high vacua is the production of pure gases. It is well known that the purity of gases plays

a very important part in all experiments where ionisation chambers and counters are used which are filled with gases at higher pressures. For example the presence of small amounts of electro-negative impurities like oxygen, chlorine, nitric oxide or of radicals of hydro-carbons shortens the life of free electrons considerably because negative ions are formed. The presence of such impurities in counters leads to a considerable distortion of the current pulses. Another example of the adverse effects of these impurities is their influence on the value of the ionisation coefficient. When electrons move through a gas in a uniform electric field, a certain number of electrons and ions are produced along each cm. of the path in the direction of the field due to collisions of electrons with gas molecules. If the field, and hence the mean energy of the electrons, is low, the ionisation coefficient is small, but since the speed of the electrons is also low, electrons become attached more readily to molecules of impurities which may be present. This is the reason why the ionisation coefficients in weak fields (low X/p) and very pure gases were recently found to be much higher than earlier observations have shown. On the other hand, the breakdown fields for gases, the molecules of which attach electrons and form negative ions or the radicals of which bind electrons, are very much higher than the breakdown fields for gases which do not form negative ions, especially, if the pressure reaches higher values.

Returning to the question of purity, a new method of preparing very pure helium has recently been developed by M. A. Biondi[16]. It has been claimed that it is possible in this way to obtain a purity of one impurity atom per 10^{10} helium atoms. This means roughly an improvement by a factor 10^5 compared with known methods. The new method makes use of the fact that liquid helium cooled to a temperature below 2.2°K transforms into helium II which has zero viscosity. Helium II is thus able to creep through minute cracks and openings e.g. those which exist when a wire is sealed into a glass capillary. Helium II, though it looks superficially like a liquid, behaves in many respects like a very dense gas. The method of obtaining this kind of very pure helium is to evacuate first of all the vessel to be filled with pure helium gas of atmospheric pressure by means of an ordinary diffusion pump, then to bake the metal vacuum system as a whole, and finally, after the valves have been closed, to use the ion gauge as a pump.

Fig. 6 shows, at the end of the vacuum system, an immersion tube which contains a platinum wire sealed into Pyrex glass serving as a link between the small upper and the lower chamber. After baking, the tube leading to the lower chamber is sealed off at A and the immersion tube is placed in liquid helium. The pressure of the helium bath is now reduced by a rotary pump to a value below 38 mm. Hg thus reducing its temperature to 2.1°K. Then the tip of the lower chamber is broken by pulling a thin metal wire, and helium II can now enter the lower chamber and diffuses along the wire into the upper chamber. In the case of a 5 mil. platinum wire which was sealed into the capillary

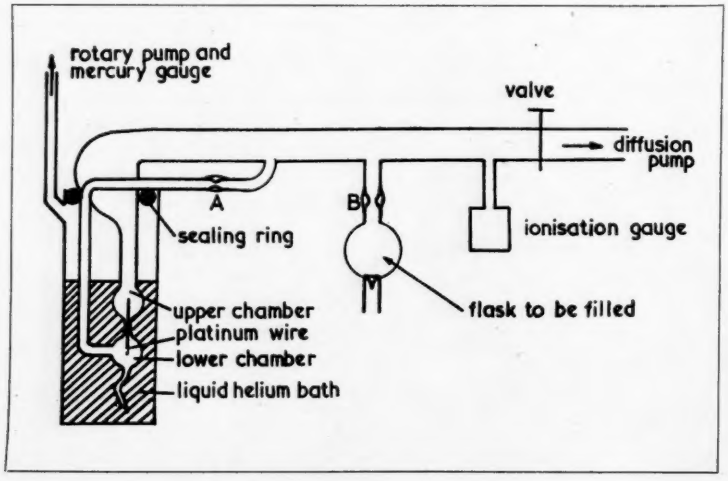

Fig. 6. Diagrammatic view of a system for the preparation of very pure helium from liquid helium II.

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for a length of half a centimetre, it took about 15 minutes to fill the upper chamber, which had a volume of 3 cm.^{3}. The viscosity of ordinary helium however, is so high that only about 10^{-4} cm.^{3} could pass through such a leak per minute. After filling the upper chamber the pumping of the helium bath was interrupted, the helium reservoir was warmed to about 4°K in order to evaporate the helium which had entered the upper chamber. As a result, the flask shown in Fig. 6 gradually filled up with helium to atmospheric pressure and could subsequently be sealed off at B.

When the constriction is sealed off, the pressure in a volume of approximately 1 litre rises by $\Delta p = 10^{-7}$mm. Hg. Since the pressure of the pure helium gas is of the order of 10^3mm. Hg, the impurity ratio is 10^{10}. It is an important factor in this connection that the impurity ratio can be kept low for a considerable period (weeks) provided the vessels have been evacuated in the manner indicated above.

CONCLUSIONS

One might ask what future developments in the field of ultra-high vacua are likely to be.

There is first of all the ion pump which does not seem to have been investigated thoroughly. Little is known about its potential applications, whether for instance, it can be used only in the pressure range where the mean free path of molecules is larger than the size of the vessel.

The fact that we can now obtain vacua which are many orders of magnitude lower than before may stimulate experiments which imitate the conditions encountered in and above the ionosphere.

Another question which requires careful investigation is the maintenance of ultra-high vacua with the help of getters. Hardly anything is known as to whether getters are of any use at extremely low pressures.

Research in nuclear physics might profit by these developments, if it were possible to construct ion pumps with pumping speeds high enough to maintain the pressure in high energy particle accelerators at levels still lower than those practicable today.

Finally, an extension of the low pressure range might be of great benefit to surface chemistry.

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KNUDSEN GAUGES

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Summary

A BRIEF review of the development and applications of the Knudsen gauge is followed by a detailed description of improved forms of metal and glass gauges. Their performance with respect to factors such as outgassing, methods of suspension, extension of range by electromagnetic compensation, and damping are discussed. The theory of the absolute manometer is reviewed. In particular, the fundamental assumptions concerning accommodation coefficient, the edge effect due to large heater-to-vane distance, and vane temperature, in the low pressure (Knudsen) region are discussed. A theory is developed to give a correction factor for a rectangular vane and heater valid for any dimensions and distance. It is shown that under certain conditions calibration is independent of the temperature of the vane.

Sommaire

BREF COMPTE rendu sur le développement et les applications de la jauge de Knudsen, suivi de la description détaillée de formes améliorées de jauges métalliques et en verre. Discussion de leurs performances par rapport a des facteurs tels que dégasage, méthode de suspension, extension de la capacité de mesure par compensation électromagnétique, et amortissement. Revue de la théorie du manomètre absolu ; en particulier, les hypothèses concernant le coefficient d'accomodation, l'effet d'arrête dû à une grande distance palette-chauffage, la température de la palette dans la domaine des basses pressions (dites de Knudsen) sont discutées. L'on développe une théorie donnant un facteur de correction pour une palette et un chauffage rectangulaire, valable pour toutes les dimensions et distances. Il est démontré que dans certaines conditions l'étalonnage est indépendant de la température de la palette.

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INTRODUCTION

MARTIN KNUDSEN (1910) constructed a so-called 'absolute manometer' based on the principle of thermal molecular pressure[1,2]. He considered an arrangement of two plates placed a small distance apart in a highly rarified gas. If one of these plates is heated to a temperature above the surrounding walls, then the other is repelled by a force which, under certain conditions, is proportional to gas pressure. The heated surface is usually fixed on one side of a movable, lightly suspended vane. The force on the vane can be determined and is a measure of the gas pressure : this is the basis of all practical forms of Knudsen gauges.

The repulsive force on the vane as a function of the temperature of the heater and gas pressure was determined by Knudsen by considering the momentum exchange of the bombarding molecules with the vane. At very low gas pressures where the mean free path is large compared with the dimensions of the apparatus, the repulsive force is due to the greater momentum imparted to the vane by molecules coming from the heater as compared with molecules coming from the surrounding walls. We will follow other authors by denoting this repulsive force by the term 'Radiometer Force,' but it should be noted that there is otherwise no analogy with Crooke's Radiometer*. Knudsen showed that if certain simplified assumptions were made the radiometer force 'F' per cm.^{2} is given by :

$$F=\frac{1}{2}p\left(\sqrt{\frac{T_1}{T_0}}-1\right), \qquad (1)$$

* M.S. received October, 1951.

* In the Crooke's Radiometer, vanes blackened on one side were suspended in an evacuated chamber and were shown to have forces exerted on them when illuminated with radiant energy on the blackened side. These phenomena are different from the Knudsen case because they take place at much higher pressures. As postulated by J. C. Maxwell[3] the effect is an edge effect due to the 'streaming' of the gas and not a surface effect as in Knudsen's case (see also L. B. Loeb 364-386[4]). A similar effect occurs in a Knudsen gauge at higher pressures as mentioned below.

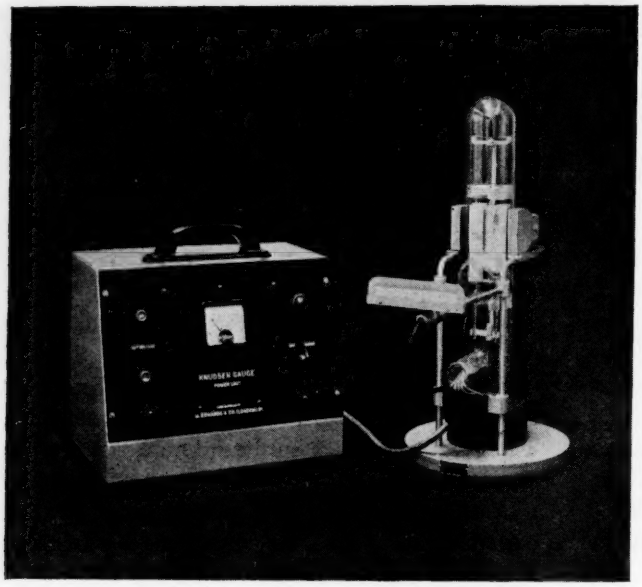
where p is the pressure of the gas to be measured in the vacuum chamber, T_1 is the temperature of the heater and T_0 the temperature of the vane and the surrounding walls. Knudsen constructed an instrument with which this formula was verified experimentally over a limited range.

Since Knudsen's early paper several attempts have been made to deduce formulae for the repulsive force between two plates under more general assumptions. These aspects will be discussed in greater detail in the theoretical section below. We will note here, however, that it was realised that in practice one or other of Knudsen's assumptions might not hold, although Knudsen gauges have been successfully constructed which give a convenient measure of pressure in the range 10^{-2} to 10^{-8} mm. Hg.

These gauges could not be classed as absolute manometers as was Knudsen's original intention but they have various important and attractive features. As may be seen from the formula (1) above, the calibration of the gauge should be independent of the kind of gas in the gauge, so that the true total gas and vapour pressure would be measured. It has been verified experimentally that it is possible to construct gauges for which this is approximately true (this point will also be discussed in more detail below) and there are no other high vacuum gauges with this important property. The dependence of the radiometer force on pressure is linear and the calibration which depends only on the temperatures and method of suspension of the vane is quite stable with proper construction of the instrument.

Various interesting Knudsen gauges have been constructed and described in the literature since Knudsen's first gauge[5-11]. Most of these gauges were not really intended as practical tools for the vacuum technician, the gauges themselves being the objects of research. The credit lies with DuMond and Pickles (1935) to be the first to point out that a more robust form of a Knudsen gauge, not itself of the absolute type, might with advantage be made[12]. They showed that a gauge of this type would be superior to other types of vacuum gauges for large metal dynamic vacuum systems, which were then becoming increasingly important for experimental physics. It was not long before instruments of this type became commercially available. These gauges were of metal construction intended for operation in the pressure range of 10^{-3} mm. Hg to 5×10^{-6} mm. Hg. Due to the nature of the work for which they were designed they were not meant to be outgassed, the heaters operating at a fairly low temperature (less than 100°C.) so that there was no danger of cracking organic vapours. These merits, as well as the general advantage of the Knudsen gauge, as discussed above, have won these instruments a place of increasing importance in vacuum work, though for industrial use there were some very serious defects. The gauges are of the suspended-vane type using a single ended torsional suspension and are still very delicate. Due to the lack of air damping, eddy current damping of the vane by means of a magnetic field has to be used, but even then the gauges have to be mounted on vibration-free supports. Other disadvantages for industrial use are : the gauges are not remote-reading, the pressure range is narrow (due to the limited length of linear scale), and the range is changed by varying the heater temperature. This last results in a restricted number of ranges and a lag in the reading after range switching. These difficulties can be largely overcome by extending the range by applying some form of compensating torque to the system as discussed in detail below. Electromagnetic compensation has been used in different ways by Fredlund[13], Klumb and Schwarz[14], and the author. An energised coil, carried by the suspension system, has been suggested by Blears[15]. Remote reading instruments can then be simply obtained by converting the deflection or mechanical displacement into an electrical signal (e.g. capacity change[16], or signal from photo-cell[15]), which is applied to a feedback system and amplifier.

Since the paper of DuMond and Pickles there has been a constant interest in Knudsen gauges. A paper describing a variant of this gauge[17], and also a number of papers dealing with the deflecting-vane type[18-25] have been published since. In the latter a thin foil strip is fixed at one edge from which it hangs down vertically. It is deflected by bending due to the radiometer force and its position observed by a



The photograph shows an experimental glass Knudsen gauge.

microscope. None of these gauges really fulfills any of the requirements of an industrial instrument as set out above.

The next important development is due to Klumb and Schwarz (1944) who also drew attention to the inadequacy of low pressure measurements when using ionisation gauges[14]. The Knudsen gauge described by them has some similarities to a construction by Riegger (1920) but is more practical and stable[10]. Both types are characterised by their symmetrical construction which ensures that the magnitude of the radiometer force remains independent of the movement of the gauge element, i.e. the sensitivity is independent of the deflection. In the Klumb and Schwarz instrument the heated surface is an inner cylinder, concentric with which is suspended a cylindrical body which in turn is surrounded by the cylindrical cold walls of the containing envelope. The radiometer body has vanes attached to it, as on a paddle wheel but at an angle to the radius. These surfaces receive the radiometer force and the consequent turning moment is measured. This construction has been examined theoretically in a paper by Weber (1947)[26]. The pressure range of the mirror and scale-type of instrument is necessarily limited. To extend the scale to higher pressures a magnetic compensation device is used. This is accomplished inductively by fixing a short-circuited coil to the radiometer body, and subjecting it to the low frequency (e.g. mains) alternating field of an external electro-magnet. The same coil may be used for damping by superimposing a constant field. They tested experimentally several instruments of this type over the pressure range 10^{-6} to 10^{-2} mm. Hg (and even higher in the non-linear region) using different methods of suspension and construction. Investigations for various permanent gases showed that indications are practically independent of the nature of the gas in the manometer.

Industrial vacuum systems are really of two fundamentally different types. In the one case there is the large volume metal system containing and dealing with a quantity of organic and other vapours. For this sort of system DuMond and Pickles had in mind their Knudsen gauge. Since there are considerable surface areas of metal in this kind of vacuum system, one may have a metal gauge which is constructed with no intention of outgassing. In an improved form of such a commercially available gauge, with the development of which the author has been associated, some of the above mentioned difficulties are overcome and will be described in detail below.

The other case is the all-glass vacuum system, the greater part of which is degassed thoroughly by baking at the highest possible temperature. This is the kind of system used in the electronic industry and for some specialised research. In both cases the accurate measurement of pressure is becoming increasingly important. The amount of metal present in the measuring apparatus must be small and if possible subjected to degassing at a higher temperature. Klumb and Schwarz had this kind of vacuum system in mind when they discussed the

superiority of the Knudsen type gauge in their paper. In another paper appearing at the same time Schwarz[27] showed to which extent the ionisation gauge, which has been almost exclusively used for this work, is fundamentally unreliable. The type of Knudsen gauge which would be suitable for these glass systems is probably the form of gauge which Wagener[28] had in mind for research on getters and similar gas exchange problems. Another obvious and important application for which such a gauge would be of use is the extension to low pressures of the measurement of vapour pressures as demonstrated by Filosofo[29]. There is no doubt that a gauge of the Klumb and Schwarz type lends itself to a construction in glass with a minimum of metallic parts. A simpler, and inherently more sensitive construction, based on the design of the all-metal gauge which meets the requirements for industrial glass systems has been constructed by the author and will also be described.

NEW KNUDSEN GAUGES

Metal Knudsen Gauge

The gauge was developed taking into account the requirements for a robust industrial gauge as mentioned above. The whole gauge assembly is easily removed from the cylindrical metal envelope to which it is secured by two lugs. The electrical supply for the heaters is brought through the envelope by glass-to-metal seals. A plane glass window is sealed into the envelope. The construction of the heater and vane assembly is shown in Fig. 1. It was not found easy to obtain an efficient directly heated surface for the gauge heater and so an indirectly heated thin-wall copper cylinder of rectangular cross section containing an axial nickel chrome heating spiral was used and found to be very satisfactory. The suspension is mounted between two flat spiral springs to safeguard it against damage by mechanical shock. The position of the lower spring is adjustable for tensioning ; the upper one can be rotated by an external control through a vacuum seal for zero setting. Various thin metal strips or wires have been found suitable as the actual suspension elements. The vane is prepared from a thin sheet of some suitable non-magnetic material ribbed for rigidity. A central rod attached to the vane carries the mirror and its two ends are joined to the suspension. The vane is thus firmly held between the two springs. In practice it is necessary to take great care that the suspension element takes off centrally from the rod, otherwise the system may be heavily unbalanced. For small balancing corrections, tiny adjustable weights are attached to a crosspiece on the vane.

To obtain the necessary degree of damping an external permanent magnet was fixed around the envelope to give a field in a direction lying in the plane of the vane.

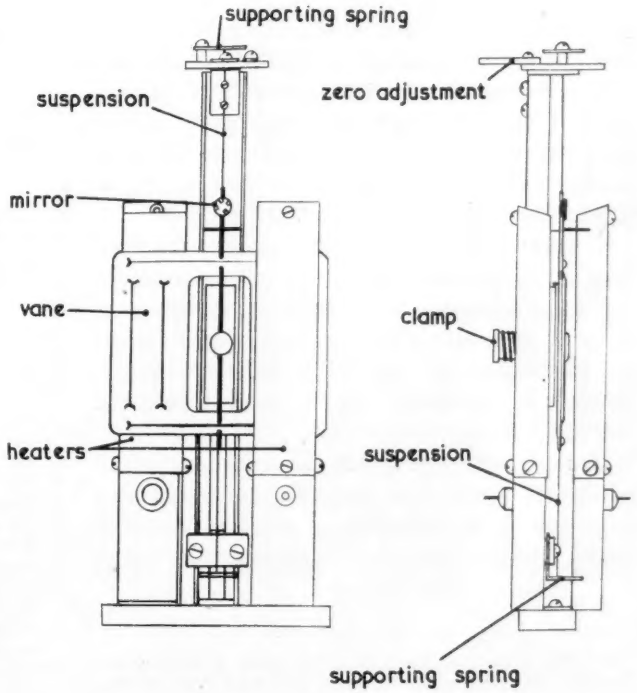


Fig. 1. Diagram to show the suspension and the construction of the vane and heater assembly of the metal Knudsen gauge.

To read pressures the usual arrangement of lamp and scale can be used (see Fig. 2). Typical calibration curves of pressure against deflection for two heater temperatures are given in Fig. 3.

Glass Knudsen Gauge

The object was to provide an instrument with a higher sensitivity which could be outgassed by baking at 400—500°C. and give, if possible, robustness and low vibration sensitivity of the metal gauge.

The whole assembly was again based on an arrangement similar to that of the metal gauge, as shown in Fig. 4, but had to be located in the glass envelope by a stainless steel spring. The heater arrangement was identical with the metal gauge, electrical connections being brought to a pinch in the side of the glass tube. To prevent distortion of the light spot by the curvature of the glass envelope, a plane glass window was sealed into it.

Metal suspensions, including tungsten, nickel-chrome alloy and stainless steel showed creep and annealing during the heating for outgassing causing permanent distortion of the wire. A solution was found in the use of quartz fibres which were found to have the required stability under these conditions. The suspension fibres were directly fused to two quartz T-pieces serving as supporting springs and at their other ends to quartz T-pieces fixed to the vane. Rotation of the upper spring for zero setting is transmitted magnetically through the envelope.

The quartz fibre construction insulates the vane from the support and heaters. To prevent therefore the vane acquiring an electrostatic charge it is necessary to sputter or evaporate a thin metal film on the suspension. An electrically continuous film is ensured by metallising the T-pieces before the suspension is assembled.

Gauges constructed in this way retain most of the advantages of the metal gauge without introducing too many of the difficulties usually associated with quartz fibre technique*. Calibration sensitivities of the required order are easily obtained using fibres 10–30 μ diameter.

DESIGN CONSIDERATIONS

Suspension and Vane

For given dimensions the sensitivity of a suspension material is governed by its modulus of rigidity ; the lower this value, the greater is the deflection for a given torque on the vane. To obtain a high sensitivity it is desirable to make the sectional dimensions of the suspension fibres or strips as small as practicable. The form of suspension in which the torsional rigidity is reduced, while the cross-sectional area is not changed, is obviously advantageous and is achieved by using a thin strip rather than a wire type of element. In practice phosphor bronze strip has been found suitable for the metal gauge and fused quartz fibre for the glass gauge as mentioned above. The approximate torsional data for a suspension can be obtained from the formulae for the applied torque ' P ' per radian deflection :

$$P = Gab^3/3L \text{ dyn. cm. for the strip } \overset{a}{\square}\, b \qquad (2)$$

and

$$P = G\pi r^4/2L \text{ dyn. cm. for the wire } \overset{2r}{\bigcirc} \qquad (3)$$

where L is the length of the suspension (cm.)
G is the rigidity modulus (dyn./cm.^{2}).

Approximate values of G for a number of useful materials are listed below, together with their ultimate tensile strengths :

Materials	Rigidity Modulus (dyn/cm^2)	Ultimate Tensile Strength (tons/in^2)
Silver	2.7×10^{11}	15
Phosphor Bronze	4.0×10^{11}	60
Beryllium Copper	4.7×10^{11}	90
Tungsten	12.0×10^{11}	70
Nickel Chromium (80/20)	7.0×10^{11}	45
Quartz	4.8×10^{11}	45

* For details see the interesting chapter in Strong's book, Ref. 31. For recent data on quartz fibres see Refs. 32, 33.

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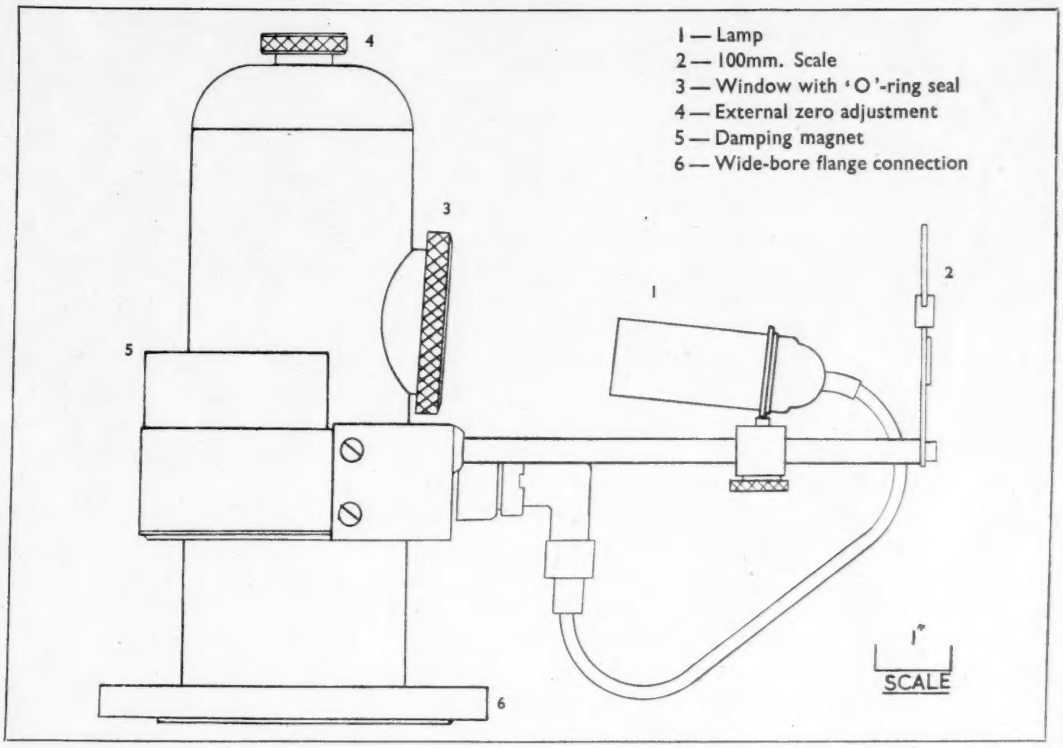

Fig. 2. Outline diagram of metal Knudsen gauge and its optical assembly.

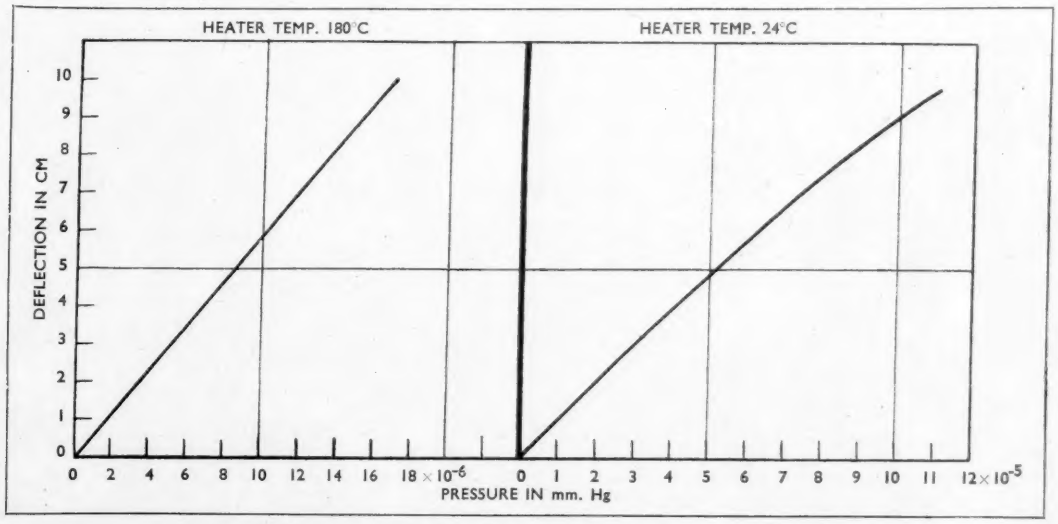

Fig. 3. Typical calibration curves of metal Knudsen gauges for two different heater temperatures (deflection plotted against pressure, scale distance 20 cm).

The values for the metals quoted above must only be taken as a guide, since the actual value obtained for a particular specimen will depend on its past history. The value of G quoted for quartz is that given by Eberhardt et al.[32,33]

In practice, the value of P for any given suspension is determined experimentally from the period of oscillation of a suspended system of known moment of inertia.

In using a double-ended suspension the mechanical stability of the gauge is much improved, though a single-ended suspension of the same total length would have a four-fold greater sensitivity.

The gauge dimensions are basically governed by the vane and suspension dimensions. Since the sensitivity of the gauge is directly proportional to the area of the vane and the length of the suspension, the optimum condition is achieved when the vane length and total suspension length are equal, with equal lengths of suspension top and bottom.

There is, however, a limit to the size of the vane and, in fact, the size of the whole gauge. One limiting factor is the mass which the suspension strip can safely carry. The thickness of the vane material is 0.001 inch and further reduction of this dimension leads to difficulties. Since the rigidity of the vane is obtained entirely by ribbing, foil thinner than 0.001 inch tends to rupture when pressed.

Ferrous impurities in the vane will cause a reduction in sensitivity due to the presence of the magnetic damping field. Worse still, appreciable changes of zero may occur due to a permanent magnetisation of the suspended system. When the vane is deflected through an appreciable angle, the direction of its permanent magnetisation alters. Thus, when the vane returns to its position of rest, the direction of magnetisation does not lie along the lines of force due to the permanent magnet. The vane thus remains deflected through a small angle. After a period of time, if the vane is not deflected again, the direction of its magnetisation gradually changes so as to lie more and more in its original direction. The phenomenon has been observed with several impure vanes and it is therefore essential to obtain materials of the highest purity and to handle them with non-ferrous tools. Thus, a gauge made from very pure foil was calibrated and then deliberately handled and cut with steel tools. On re-calibrating it showed a decrease in sensitivity of over 50%. This effect was further enhanced by increasing the damping field from 150 to 400 gauss when the contaminated gauge showed a decrease in sensitivity of over 70%. This provides a convenient and easy check of the ferrous contamination of a gauge.

Damping

The taut, double-ended method of suspension, allows the vane to move mainly by rotation. Owing to the absence of gas friction the vane would swing freely with the slightest mechanical disturbance ; it is therefore necessary to provide powerful damping. As mentioned above, the vane is subjected to a magnetic field, and damping can be considered as being due to the eddy currents set up in the vane, which acts as a short-circuited coil of area A.

To work out the magnetic field H required for critical damping of the vane, we note that if θ is a small angular deflection of the vane, the differential equation of θ is given by :

$$\frac{d^2\theta}{dt^2} + \frac{H^2A^2}{rI}\frac{d\theta}{dt} + \frac{c\theta}{I} = 0, \qquad (4)$$

where r is the resistance of the coil, I the moment of inertia about its axis of suspension, and c the restoring torque. Converting to practical units, the condition for critical damping is :

$$H^2 = \frac{2r\sqrt{cI}}{A^2} \times 10^9, \qquad (5)$$

H being given in gauss and r in ohms. In terms of the density δ, electrical resistivity ρ, and time constant T, this may be written as

$$H^2 = 32\,\pi\,\delta\rho \times \frac{10^9}{3T}. \qquad (6)^*$$

* It should be noted that there is a slight discrepancy between this result and a formula given by DuMond and Pickles (Ref. 12 p. 367).

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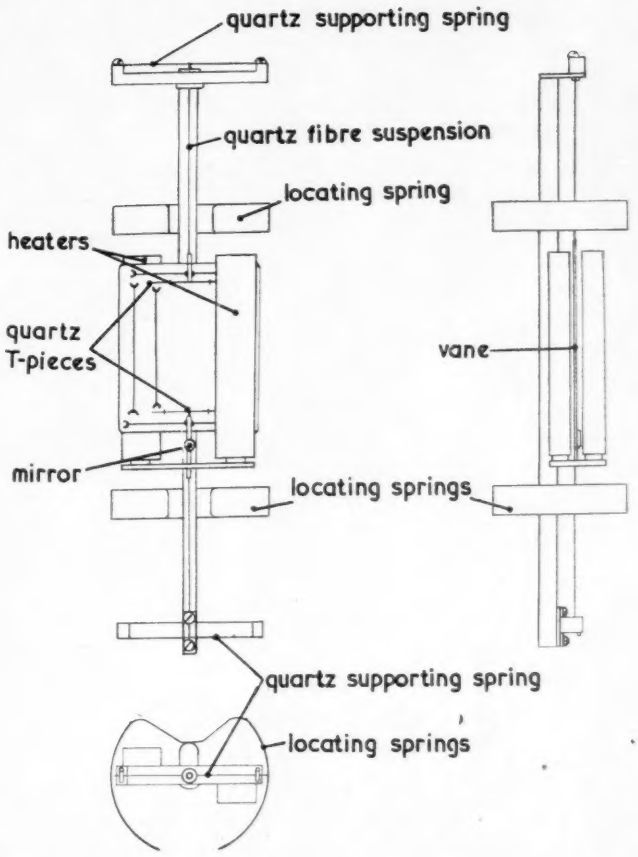

Fig. 4. Diagram to show the method of suspension of the vane and the heater assembly of the glass Knudsen gauge.

It is easy with modern permanent magnet materials to obtain the magnetic field for critical damping, in spite of the necessarily large air gap. A field of 120 to 150 gauss was found adequate for our systems.

Pressure Range

The lower limit of the pressures which can be measured with a Knudsen Gauge is set by the delicacy of the suspension as discussed above, and the maximum temperature difference between the heater and cold walls which can be tolerated. The limit of the latter is set by degassing and other considerations, usually to a few hundred degrees Centigrade. A further extension is achieved by cooling the outside of the gauge envelope, but this is often not practicable. The optical magnification usefully employed on any gauge depends on the zero stability and residual (higher frequency) vibration of the system. These factors are determined by the purity of the vane, method of suspension and mounting of the gauge. The author found in practice that there was no difficulty in obtaining sensitivities of the order of 10 cm. per 2.5×10^{-5} mm. Hg with heater temperatures of 200°C. and scale distances of 30 cm. when using very robust suspension systems. Sensitivities of ten times this value were easily obtained with slightly more delicate suspensions.

Once a given suspension has been chosen, the total pressure range of the instrument used in the ordinary way, is very limited. The scale length for a given scale distance is necessarily limited by the maximum angular deflection. The sensitivity can be decreased by reducing the heater temperature difference to a minimum of 10°C. for thermal stability so that the total pressure range of a gauge is only of the order of 500 to 1. Also, unless the heat capacity of the heaters is very small, it inevitably takes some time to obtain thermal equilibrium after changing the range in this way.

It has therefore been suggested that some form of electrical compensation be applied to the system to bring back the vane to its zero position, the amount of compensation being a measure of the pressure. Klumb and Schwarz have used an alternating magnetic field acting inductively on a short circuited coil specially provided on top of their vane system. In the case of the author's gauges described above this method of compensation is also applicable, the vane itself being used, as for eddy current damping. A pair of coils, with transformer stampings as the core has been found to be very effective, the current required for returning the vane to zero being calibrated in terms of pressure. In this case a direct

current coil can be used on the same magnetic circuit to obtain the necessary damping field.

It is obvious that such a method of compensation can be easily converted into a remote reading or recording Knudsen gauge by supplying the necessary feedback loop, e.g. by a photo-cell.

Using a compensated Knudsen gauge with a fairly fine suspension there remains no reason why the full possible range of the gauge should not be covered with a single instrument. We will briefly examine the theoretically possible range of such an instrument.

First consider the lower limit. The radiometer force is proportional to pressure for given values of heater and gauge temperatures and will steadily decrease with pressure ; but due to the fixed heater temperature, there will act on the vane a constant pressure due to the electromagnetic radiation from the heater. This is given by :

$$\frac{4}{3}\frac{\sigma e}{c}(T_1^4 - T_0^4) \qquad \text{dynes. cm}^{-2}, \quad (7)$$

where e is a factor due to the emissivities of the surfaces considered, σ is Stefan's constant and c denotes the velocity of light. An approximate calculation of this effect, assuming e=1 and using Eqn. (1) for the molecular pressure, can be made. It is then found that when the gas pressure is sufficiently reduced, the molecular pressure is eventually comparable with, or less than, the radiation pressure. Thus the lower limit at which the molecular pressure still exerts a useful proportion of the total pressure on the vane for purposes of measurement is probably 10^{-8} mm. Hg. Some representative figures are given in tabular form.

Heater Temperature (above ambient) °C.	Gas Pressure mm. Hg	Radiatn. Pressure %	Molec. Pressure %
10°C.	10^{-6}	6	94
	10^{-7}	37	63
	10^{-8}	86	14
60°C.	10^{-6}	7	93
	10^{-7}	44	56
	10^{-8}	89	11
100°C.	10^{-6}	9	91
	10^{-7}	50	50
	10^{-8}	91	9

This indicates that no useful purpose seems to be served in attempting to reduce the low pressure sensitivity of the gauge much below 10^{-7} mm. Hg.

It should be noted that if the envelope temperature is decreased, then for the same temperature difference the molecular pressure is increased while the radiation pressure is reduced, so that an extension of range is achieved ; this is usually impracticable since the gauge would then act as a cold sink for the vacuum system.

Now consider the upper limit. The calibration of the Knudsen gauge is linear with increasing pressure up to a point, at which the mean free path is of the same order as the physical dimensions of the gauge. Above this the rate of increase of deflection diminishes and at a certain pressure a maximum deflection occurs. These phenomena and the dependence of the maximum on the various parameters of the gauge (heater-vane distance, accommodation coefficient, etc.) have been investigated by several workers[13,26,34-39]. We will here only be concerned with the gauge in its linear pressure range (the Knudsen condition), i.e. up to a pressure which was found to extend into the micron region.

These considerations show that the physically realisable range of the Knudsen gauge extends from about 10^{-2} to 10^{-8} mm. Hg which is comparable to ordinary three-electrode ionisation gauges.

Calibration

The gauges are calibrated on a simple pumping system using a high speed diffusion pump (60 litres/sec. or more) with a baffle system and cold trap. Dry air is admitted below the cold trap through a fine control needle valve. The metal gauges are connected to a metal header placed on top of the diffusion pump. A freely suspended ionisation gauge in a water cooled tube of similar diameter to the Knudsen gauge is mounted alongside. The ionisation gauge was calibrated against a high compression McLeod gauge, the calibration factor being re-checked before and after use. In fact, no discernible change in calibration was observed.

Calibrations of the Knudsen gauges above 10^{-4} mm. Hg could be done directly against the

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McLeod gauge, all lower pressure calibrations being done with the calibrated ionisation gauges.

Similar vacuum systems were used to calibrate glass Knudsen gauges. In this case a wide-bore glass ionisation gauge was included in the Knudsen gauge envelope. The whole assembly could be baked together.

It should be noted that the backing pump was independently mounted, and connected to the diffusion pump by a short length of rubber tubing ; otherwise no special precautions were taken to prevent vibration.

THEORETICAL CONSIDERATIONS

Knudsen[1,40-43] originally made the following assumptions,

(i) The distance between the two plates (heater and vane) is negligibly small relative to their dimensions.

(ii) The pressure is so low that the mean free path of the gas molecules is large compared with the dimensions of the apparatus.

(iii) The vane is at the same temperature as the surrounding walls of the apparatus.

(iv) Accommodation of energy at the walls hit by the molecules is perfect so that the mean energy of the molecules after hitting a wall corresponds to the temperature of the wall. The velocities of molecules leaving a wall are determined completely by the temperature of the wall.

Under these assumptions he was able to show that the force per cm^2 on the cold vane is given by Eqn. (1).

It was realised that in practice one or other of Knudsen's assumptions might not hold. Thus Smoluchowski (1911) showed how Eqn. (1) would have to be modified if assumption (iv) was changed to allow for the coefficients of accommodation of the two plates[44]. He developed his theory along the lines of two alternative hypotheses, which he denoted by A and B.

Hypothesis A is equivalent to that made by Knudsen[2,45]. It assumes that if molecules leave a surface, they have a normal distribution of velocities with equal probabilities in all directions of space, irrespective of the velocity distribution of the molecules striking the surface; also, when molecules of a temperature T_1 strike a surface at a higher temperature T_2, the molecules leaving the surface have a temperature T which is intermediate between T_2 and T_1 given by the relation

$$T-T_1=\alpha(T_2-T_1), \qquad (8)$$

where α is a number less than unity (called by Knudsen 'the accommodation coefficient').

Hypothesis B is similar to that originally used by Maxwell in dealing with the 'slip' of gases[3]. It assumes that of all the molecules striking a surface, a fraction f reaches thermal equilibrium with it, while the fraction (1-f) is reflected with the same velocity, i.e., temperature. According to this hypothesis the velocity of molecules leaving a surface may be different in different directions.

Under hypothesis (A) Smoluchowski deduced that Eqn. (1) should be replaced by :

$$F=\frac{p}{2}\left(1+\frac{a_1-a_2}{a_1+a_2-a_1a_2}\right) \times\left(\sqrt{\frac{T_1}{T_0}}-1+\frac{a_2(2-a_1-a_2)(T_1-T_0)^2}{4(2-a_2)(a_1+a_2-a_1a_2)T^2}\right), \qquad (9)$$

where the accommodation coefficients for the two walls are a_1 and a_2. When $a_1=a_2=a$ the equation is reduced to

$$F=\frac{p}{2}\left\{\sqrt{\frac{T_1}{T_0}}-1+\frac{1-a}{4(2-a)^2}\left(\frac{T_1}{T_0}-1\right)^2\right\}. \qquad (10)$$

Under assumption (B) Smoluchowski deduces that the Knudsen equation (1) would have a factor

$$1+\frac{f_1-f_2}{f_1+f_2-f_1f_2}$$

if the values of f for the two surfaces are f_1 and f_2 respectively. When these values are the same,

i.e. $f_1=f_2=f$, then this factor becomes 1, i.e., then the Knudsen formula holds without correction.

Smoluchowski considered that the accuracy of experimental results had not been sufficient to distinguish between these hypotheses and that in all probability the truth is intermediate between the two. Since this statement was made, much work has been done especially in the radiometer pressure region, with the result that hypothesis A is generally accepted as a good approximation. Fredlund (1932) had done experiments with plates of vastly different surface finish[13]. Calibrating his gauge with different gases, he found gas-dependent factors, which could be correlated with possible values of the accommodation coefficients. He also showed that when the surfaces had similar accommodation coefficients the deviation could be made negligibly small. This is in accord with the findings of Klumb and Schwarz*.

Martin (1930) modified assumption (i) above to the other extreme, where the distance between the plates is considered large relative to their physical dimension[46]. Martin's approximations are not justified as was shown by Spivak (1932), who deduced a correction to Eqn. (1) for the case when assumption (i) is dropped altogether[47,48]. He considered the general condition of a square vane lying parallel to a square heater with sides of equal length b and separated by the distance d. He showed that under the two limiting conditions of b large compared with d, and the other extreme of d very large compared with b, one obtains from his formula the Knudsen formula (Eqn. 1), and the corrected Martin relation, respectively. He also showed that one could allow for the accommodation coefficient.

Fredlund (1932) also considered the effect of vane distance in his investigation and worked out a formula[13,35]. He also investigated the effect of the ratio of vane size to separation on the upper limit of linearity of the pressure calibration curve.

The investigation of Spivak is of some interest because it shows how the Knudsen gauge is influenced by heater-to-vane distance. A formula was worked out by the author generalizing Spivaks method to apply to a rectangular vane and heater arrangement. It was found that there was an error in Spivaks derivation so that his formula gives the wrong result. This is due to the limits of integration in his calculation. Only the main points of the argument will be given here.

Consider the arrangement shown in Fig. 5. A heater H and vane V, taken to be rectangular plates with sides of lengths a and b are separated by the distance d. These plates are surrounded by an enclosure E. The surfaces H, V and E are supposed to have uniform temperatures T_1, T_2 and T_0 respectively.

The pressure p inside E is assumed low enough for Knudsens assumption (ii) to hold, i.e., collisions between gas molecules are neglected when considering the motion of molecules from one plate to another. It is also assumed for the moment that assumption (iv) above holds. Consider also the schematic arrangement in Fig. 6.

It is seen that the forces on the surface V are made up of momentum exchanges due to the following groups of molecules (depicted in the diagram by 1, 2, 3, 4, 5) :

1. Molecules coming from H which strike V_H (this denotes the side of V facing H).
2. Molecules coming from E which strike V_H.
3. Molecules coming from V_H.
4. Molecules coming from V_E (the side of V facing the enclosure).
5. Molecules coming from E which strike V_E.

Consider the densities of molecules (meaning number of molecules per unit volume) near the surfaces H, V and E. Let these densities be n_1, n_2, n_0 respectively. If we only consider molecules moving in directions away or towards these surfaces, the densities will be halved ; i.e., $\frac{1}{2}n_1$, $\frac{1}{2}n_2$, $\frac{1}{2}n_0$. We assume Maxwellian velocity distribution for the molecules in the groups 1 to 5 above. Consider the points P (x, y, O) on H and Q (x′, y′, d) on V respectively (Fig. 5).

Expressing (x′, y′) in terms of polar co-ordinates θ and ϕ, we have :

$$P'S=x'-x=d\tan\theta, \qquad (11)$$

* See also the discussion by Weber (Ref. 26), though dealing mainly with higher pressures.

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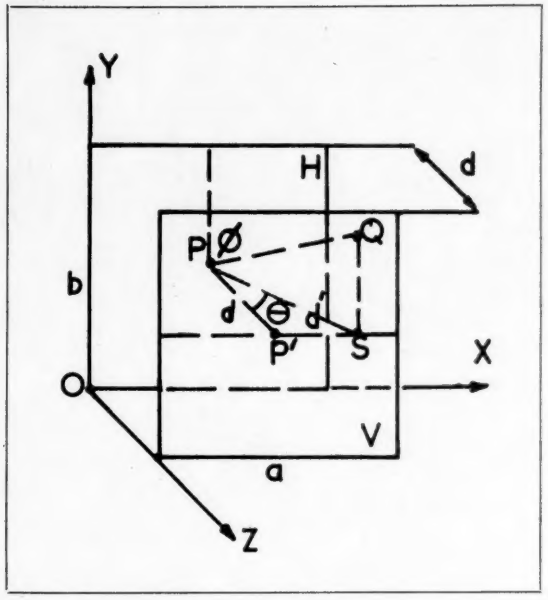

Fig. 5. Diagrammatic sketch of the heater-vane arrangement. The heater H is shown in the xy plane and the vane V is shown in a parallel plane.

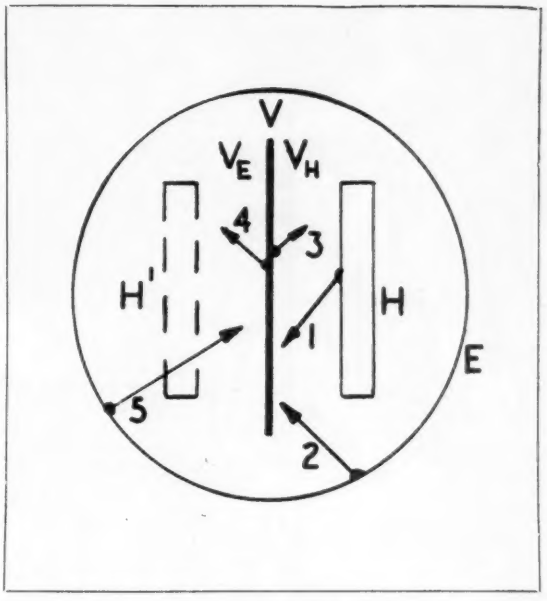

Fig. 6. Diagram of the plan view of the heater-vane arrangement and envelope in relation to the groups of molecules 1-5. The image heater surface H′ is shown by dotted lines.

and with

$$d' = d/\cos\theta$$
$$QS = y' - y = d'/\tan\theta = d/\tan\phi\cos\theta, \quad (12)$$

Using Knudsen's cosine law, the number of molecules leaving H from the element dx dy and arriving within the solid angle subtended by dx′ dy′ at P with velocities in the range (c_1, c_1+dc_1), is

$$(c_1 dn_1/4\pi)\sin^2\phi\cos\theta\, d\phi\, d\theta\, dx\, dy, \quad (13)$$

where as usual

$$dn = 4n\sqrt{\frac{hm^3}{\pi}}\, e^{-h^2 mc^2} c^2 dc, \quad (14)$$

with m = mass of molecules
n = number of molecules per unit volume
h = 1/2kT (k = Boltzmann's constant)

The momentum imparted on V by each molecule (mass m, velocity c_1) is $mc_1 \sin\phi\cos\theta$. So that the momentum exchange at V for all the molecules coming from H is :

$$\frac{m}{4\pi}\int_0^\infty c_1^2 dn_1 \int_{\phi_0}^{\phi_1} \sin^3\phi\, d\phi \int_{\theta_0}^{\theta_1} \cos^2\theta\, d\theta \int_0^a dx \int_0^b dy \quad (15)$$

taking—

$y' = b \quad \phi_0 = \tan^{-1}(d'/(b-y))$
$y' = o \quad \phi_1 = \frac{1}{2}\pi - \tan^{-1}(-y/d') = \frac{1}{2}\pi + \tan^{-1}(y/d')$
$x' = o \quad \theta_0 = \tan^{-1}(-x/d) \quad = -\tan^{-1}(x/d)$
$x' = a \quad \theta_1 = \tan^{-1}((a-x)/d).$ (16)*

Carrying out this integration, we get for the last four integrals :

$$I = \frac{4}{3}\left\{ab\sin^{-1}\left(\frac{ab}{\sqrt{d^2+a^2}\sqrt{d^2+b^2}}\right) + d\sqrt{a^2+b^2+d^2} - d\sqrt{d^2+a^2} - d\sqrt{d^2+b^2} + d^2\right\}, \quad (17)$$

* These limits are incorrect in Spivak's work.

In the special case a=b, considered by Spivak, this becomes :—

$$I_S=\frac{4}{3}\left\{a^2\sin^{-1}\frac{a^2}{(a^2+d^2)}+d\sqrt{2a^2+d^2}-2d\sqrt{a^2+d^2+d^2)}\right\}. \tag{18}$$

Also

$$\frac{m}{4\pi}\int_0^\infty c_1^2 dn_1=\frac{3n_1}{8\pi h_1}, \tag{19}$$

so that the mean pressure on the plate V due to the presence of the hot plate H (by group 1 of the molecules) is :

$$\overline{p_H}=\frac{n_1}{2h_1}\gamma, \tag{20}$$

where

$$\gamma=\frac{2}{\pi}\left\{\sin^{-1}\left(\frac{ab}{\sqrt{a^2+d^2}\sqrt{b^2+d^2}}\right)+\frac{d}{ab}\left(\sqrt{a^2+b^2+d^2}-\sqrt{a^2+d^2}-\sqrt{b^2+d^2}\right)+\frac{d^2}{ab}\right\}. \tag{21}$$

For a=b we get :

$$\gamma_S=\frac{2}{\pi}\left\{\sin^{-1}\left(\frac{a^2}{a^2+d^2}\right)+\frac{d}{a^2}\left(\sqrt{2a^2+d^2}-2\sqrt{a^2+d^2}\right)+\frac{d^2}{a^2}\right\}. \tag{22}$$

In the limiting cases

(1) d ⟩⟩ a :

$$\gamma_S\to\frac{2}{\pi}\frac{\frac{a^2}{d^2}}{\frac{a^2}{d^2}+1}\to\frac{2}{\pi}\frac{a^2}{d^2}, \tag{23}$$

(2) a ⟩⟩ d :

$$\gamma_S\to\frac{2}{\pi}\left\{\frac{\pi}{2}-\frac{d}{a}(2-\sqrt{2})\right\}=1-0.373\frac{d}{a}\to 1, \text{ as } d\to 0. \tag{24}$$

It is also instructive to work out the mean probability ' ω ' of molecules from H which will strike V. In this case one can integrate the expression above between the same limits. Carrying out the integration one obtains, by reasoning similarly to Spivack that :

$$\omega=\frac{2}{\pi}\left\{\sqrt{1+\frac{d^2}{a^2}}\tan^{-1}\frac{\frac{b}{d}}{\sqrt{\frac{a^2}{d^2}+1}}+\sqrt{1+\frac{d^2}{b^2}}\tan^{-1}\frac{\frac{a}{d}}{\sqrt{\frac{b^2}{d^2}+1}}+\frac{d^2}{2ab}\log\frac{\left(\frac{b^2}{d^2}+1\right)\left(\frac{a^2}{d^2}+1\right)}{\frac{b^2}{d^2}+\frac{a^2}{d^2}+1}-\frac{d}{a}\tan^{-1}\frac{b}{d}-\frac{d}{b}\tan^{-1}\frac{a}{d}\right\}. \tag{25}$$

If a=b we get :

$$\omega_S=\frac{2}{\pi}\left(2\sqrt{1+\frac{d^2}{a^2}}\tan^{-1}\frac{\frac{a}{d}}{\sqrt{\frac{d^2}{a^2}+1}}-2\frac{d}{a}\tan^{-1}\frac{a}{d}+\frac{d^2}{a^2}\log\frac{\frac{a^2}{d^2}+1}{\sqrt{2\frac{a^2}{d^2}+1}}\right). \tag{26}$$

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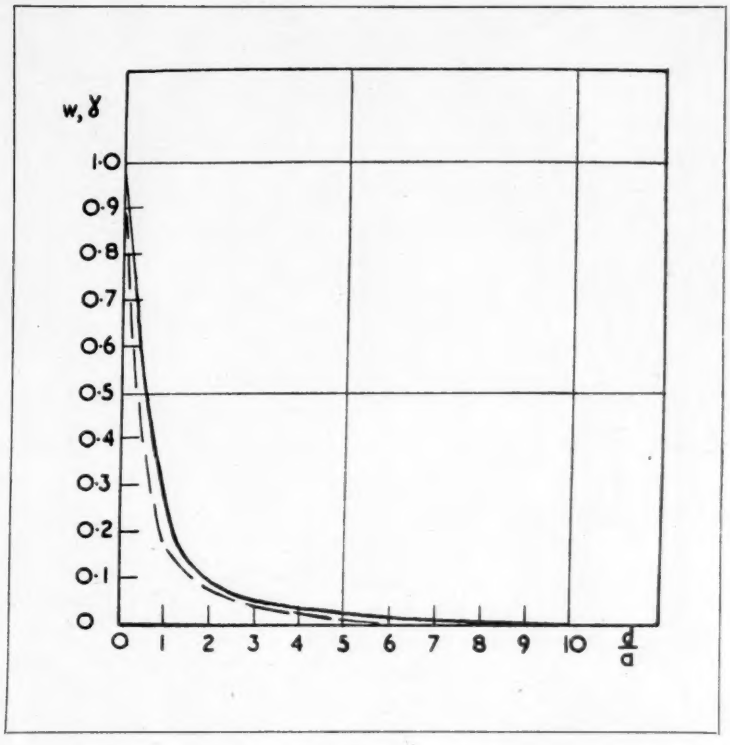

Fig. 7. The Knudsen formula correction factor γ (solid line) and the probability ω_S (dotted line) of molecules from the heater colliding with the vane, calculated for a square vane and plotted as a function of the vane separation / vane length ratio.

In the limiting case a $\rangle\rangle$ d :

$$\omega_S \rightarrow \quad 1 - \frac{4}{\pi}\frac{d}{a}\tan^{-1}\left(\frac{a}{d}\right). \qquad (26a)$$

Fig. 7 is a graph showing γ_S and ω_S as a function of d/a.

Consider now the other groups of molecules 2, 3, 4, 5, above and their effect on V. It is clear that in the case when α, the accommodation coefficient is assumed 1, the molecules in groups 3 and 4 coming from either side of V depend in their velocity distribution only on the temperature of the vane and not on their previous history, hence their mean momentum exchange with the vane is cancelled out. Now consider molecules belonging to group 5, coming from the external envelope E. If we imagine a mirror image heater H′ facing the V_E side of the vane, of the same size as H, and separated by the distance d, then we can divide the molecules from E which hit V_E into two groups : those coming from directions which would pass through the surface H′ and the remainder coming from points on E which are not ' shadowed ' by H′. It is obvious that the effect on V of the latter molecules is balanced exactly by the effect on V of molecules in group 2 (i.e. molecules falling on V_H coming from E). Hence the resultant total mean pressure on V is due only to molecules coming from H and molecules coming from the imaginary surface H′, i.e., the total mean pressure on V is

$$\bar{p} = \overline{p_H} - \overline{p_{H'}} = \frac{n_1}{2h_1}\gamma - \frac{n_0}{2h_0}\gamma \qquad (27)$$

We now seek a connection between n_1 and n_0. Note that in the steady state (i.e. in the absence of gas flow) as many molecules leave P on H as arrive at the symmetical point P′ on V so that integrating over H and V we obtain :

$$\frac{n_1}{\sqrt{h_1}} = \frac{n_2}{\sqrt{h_2}}, \qquad (28)$$

similarly for the image surface H′ and V, so that :

$$\frac{n_2}{\sqrt{h_2}} = \frac{n_0}{\sqrt{h_0}}, \qquad (29)$$

Similarly we get for the steady state for gas flow through the space between the plates and the envelope :

$$\frac{n_1}{2\sqrt{h_1}} + \frac{n_2}{2\sqrt{h_2}} = \frac{n_0}{\sqrt{h_0}}$$

This equation is just a re-statement of equation (28) and (29) which can therefore be disregarded.

Hence we finally obtain :

$$p = \frac{1}{2}\frac{n_0}{h_0}\left(\frac{n_1}{n_0}\frac{h_0}{h_1} - 1\right)\gamma = \frac{1}{2}\frac{n_0}{h_0}\left(\sqrt{\frac{h_0}{h_1}} - 1\right)\gamma.$$

But the pressure p of the gas in the system is assumed equal to that in the space surrounding the plates, given as usual by $p = n_0/h_0$, so that the force per cm² on the vane is finally :—

$$F = \frac{1}{2}p\left(\sqrt{\frac{T_1}{T_0}} - 1\right)\gamma, \qquad (30)$$

with γ as given by Eqn. (21) above.

It is seen that this equation does not depend on any assumptions about the temperature of the vane, so long as it is assumed, as above, that the accommodation coefficients of the surfaces are all 1. Fredlund (1932) derived results of a more general character which were tested experimentally as mentioned above[13]. Spivak also investigated theoretically the effect of an accommodation coefficient different from 1, but assuming the vane temperature to be the same as that of the envelope. The latter assumption is certainly not true for sensitive practical manometers. The evidence so far seems to be that provided all surfaces have a similar finish and are not too highly polished the effect of any accommodation coefficient is small ; the formula (28) derived above is then fairly accurate. It would be worthwhile to extend the experiments and investigations of Fredlund for various surfaces and different gases, but including the effect of vane temperatures.

CONCLUSIONS

Knudsen gauges of the suspended-vane type can be constructed to measure in the pressure range 10^{-2} to 10^{-8} mm. Hg. A metal gauge with phosphor-bronze strip taut suspension and employing eddy current damping was found to have the required robustness and low vibration sensitivity for use with industrial metal systems. There are many applications for which a glass gauge capable of thorough outgassing is required, and a sensitive instrument based on the metal design, but having a taut quartz fibre suspension fulfils this requirement. The sensitivity and zero stability is very much bound up with the purity of the vane material, but by scrupulous care in the assembly of the gauge this effect can be made small for the field strengths required for critical damping. The inherently narrow range of these instruments may be extended by applying a compensating torque. An alternating magnetic field acting inductively on the vane was found to be an efficient method of extending the range. The Knudsen gauges described were not intended to be 'absolute' instruments in the sense that their calibration could be calculated from Knudsen's formula. It is found however that the influence of accommodation coefficients and heater to vane distance can be estimated for these gauges by using the formulae derived in the theoretical section. When the effect of accommodation coefficients is negligible, the calibration is found to be unaffected by the temperature of the vane.

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Activation of HIGH VACUUM OXIDE-CATHODE VALVES

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Summary

THE ARTICLE discusses various aspects of the activation of high vacuum oxide-cathode valves. The three current theories of activation—chemical reduction, electrolysis and thermodynamic—are considered and discussed on the basis of the author's own laboratory experience. None of the theories are considered to give at present an adequate explanation of the high level of initial activation now common in oxide-cathode valves.

Sommaire

L'ARTICLE DISCUTE differents aspects de l'activation de tubes à haut vide à cathode d'oxydes. Les trois théories courantes d'activation—réduction chimique, électrolyse et thermodynamique—sont étudiées et discutées sur la base de l'expérience de l'auteur dans son propre laboratoire. Il est conclu qu'aucune des théories ne donne actuellement une explication adéquate du niveau élevé de l'activation initiale, maintenant courante en tubes avec cathodes à oxydes.

INTRODUCTION

THE PHYSICAL MECHANISM of activation of oxide-cathodes is a mystery which has intrigued engineers and physicists for a quarter of a century. A major industry, basically dependent on the phenomenon, has put forward great effort towards a solution but even today has succeeded only in controlling its course rather than understanding its mechanism. Such a reliance on empiricism is unacceptable and efforts towards a solution continue on both sides of the Atlanic.

The present article is neither a review nor yet a research report ; it is a frankly personal account of some work on cathode activation which is being carried out intermittently in one corner of one laboratory in England. The treatment is frequently dogmatic and the general approach that of an informal interim report to a brother engineer who can be relied upon to sort fact from speculation.

The article opens with some purely factual information concerning the comparative emission levels achieved in valves processed under industrial conditions and under conditions of strict laboratory control. This study gives some idea of the degree of success achieved in industry in initiating and maintaining a high level of activation. The remainder of the article deals with the activation problem itself – largely from the experimental standpoint.

EMISSION LEVELS

Activation of the cathode is achieved by building into the oxide matrix an excess of metallic barium. As a result of this excess metal element the heated cathode is able to conduct and emit, and the level of emission attained at any particular temperature is regarded as a function of the concentration of excess barium in the matrix.

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A start will be made with a few words about the assessment of emission level. The emission measured must of course be total emission and to have any significance it must be coupled with the area and temperature of the cathode surface. Two methods of measurement are commonly available ; the first a high voltage pulse method for use at the normal operating temperature (1,050°K) of the cathode and the second a simpler d.c. method for use at low cathode temperature (circa 700°K). The pulse method is invaluable in determining absolute values of emission at normal temperatures whilst the low temperature technique is perhaps the more useful for comparing emissions of valves of similar structures and for detecting changes in emission in a particular valve. Figures quoted in this section are all taken by the low temperature method at a nominal 700°K – equivalent to about 400 milliwatts in a 2-watt cathode.

The low temperature total emission of such a typical pentode valve as the CV 138 may be measured by lowering the cathode power from 2.0 watts to an accurately adjusted 400 milliwatts and then measuring the total emission current drawn to the positively-primed control grid. The cathode power of 400 milliwatts in a 2-watt cathode represents a nominal temperature of 700°K at which the total emission may be expected to be in the range 0.10 mA to 7.0 mA. Measurement of a group of samples of CV 138 drawn from the same day's run in a factory show a wide spread of total emission values. Such a spread might at first thought be put down to uncontrollable factors inevitable in high-speed mass production, but such does not in fact appear to be the case. Work over a period of years at Dollis Hill has shown that the same, or even a wider spread occurs with valves which have been prepared, assembled and processed in apparently identical fashion under the closest conceivable laboratory control. A few facts and figures concerning a particular run may help in illustrating the phenomenon. The valve used was the G.P.O. Standard Diode 6D10 which is identical mechanically to the cathode and control-grid structure of the commercial pentode CV 138—it was indeed so chosen to enable direct comparison to be made of emissions obtained in the Dollis Hill laboratories with those of commercial firms making the CV 138. In the test batch of 112 diodes the utmost care was given to pre-processing of the piece-parts for cleanliness and the cathode paste was obtained from a single supply bottle. All valves were pumped on a high-grade bench pump with a manifold pressure of $\angle 2\times10^{-6}$mm. Hg. Total emission measurements were made by applying 400 milliwatts to the cold cathode and noting the current in milliamps drawn to the grid-collector primed at +5.0 volts. Results are set out in Fig. (1a) in the form of a pillar graph. It is apparent that the mere exercise of a close laboratory standard of control is quite insufficient to control the spread which must be due to factors not yet understood.

Whilst it has so far proved impracticable to control emission spread it has been found possible to influence the *average* level of emission in a marked manner. It has previously been mentioned that the G.P.O. Standard Diode used in Fig. (1a) is identical in G1-cathode structure of the pentode CV 138 and direct comparison of total emission at 400 milliwatts is therefore possible. Fig. (1b) gives pillar graphs of the total emissions of batches of commercial CV 138 drawn from various manufacturing sources. Comparison of Fig. (1a) and (1b) shows that both spread and absolute level of emission is much higher in the case of the laboratory-processed diodes than it is in the case of the groups of commercial samples. The reason for this result is clear : the absolute level of emission achieved is dependent on the efficacy of piece-part pre-processing and pumping and the lower average levels of the commercial groups are due to excess barium oxidation or gas 'poisoning' at a late stage in the production process. An interesting point now arises in regard to the spread of the laboratory-processed group. Is this due to varying degrees of self-poisoning or does the mechanism for producing excess barium, on which the emission level depends, vary in drastic fashion amongst samples within the group ? In view of the care taken to achieve uniformity in the preparation of the diodes it seems logical to assume that all are subject to the same degree of poisoning and that the spread is therefore due to variation in efficiency of the barium-

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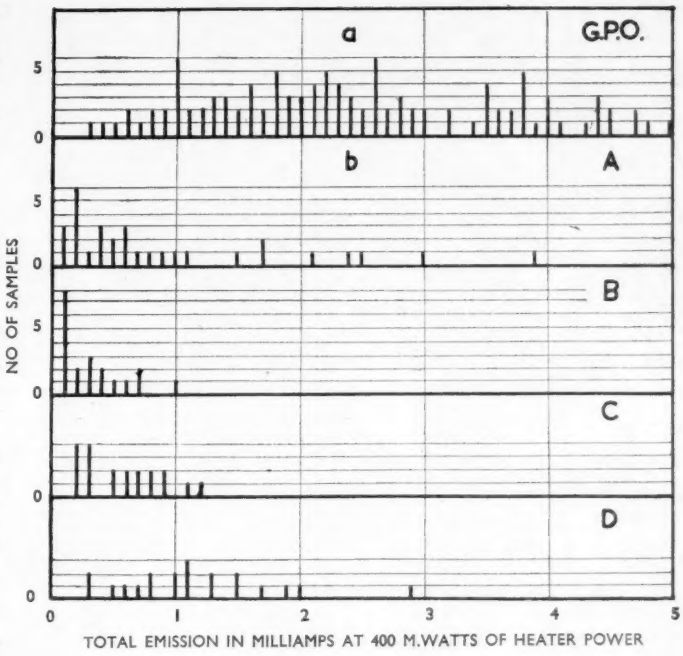

Fig. 1. Variation of emission levels: (*a*) refers to 109 samples of G.P.O. valves (*b*) refers to valves made available from a number of manufacturers. Manufacturer (A) : 31 samples. Manufacturer (B) : 20 samples. Manufacturer (C) : 20 samples and Manufacturer (D) : 20 samples.

producing mechanism. If this view is accepted it seems that the average level of emission of a co-produced group is determined by the average degree of poisoning to which the group has been subjected during processing ; and the spread within the group is due to variation in the efficacy of the barium-producing mechanism.

It now seems opportune to turn to the difficult question of the mechanism of excess barium production. A first possibility arises from the thermo-dynamic theories of Schottky, Frenkel, Jost, etc. The application of these theories to the oxide-cathode has had great success in explaining the phenomenon of electronic conduction and there seems no reason why the initial activation cannot also be covered. If it is assumed for example that lattice vacancies of both ion sorts are necessary for thermal equilibrium at any temperature, then we only require some agency to remove one sort to leave an excess of the other. The means for removal might be chemical or result from a current flow.

Other possibilities for excess production have been reviewed by Herrmann and Wagener[1]:

(a) Direct chemical reduction of barium oxide by some reducing agent, e.g. Mg or Si from the core metal, carbon from the matrix binder, or carbon monoxide generated during the processing treatment.

(b) Electrolytic production of barium involving faradaic evolution of oxygen from the BaO matrix.

These various theories will be examined in greater detail.

CHEMICAL REDUCTION THEORY

The reason for using cathode cores of nickel containing small quantities of reducing agents is a simple and sound one – industry has found over a period of years that it is far easier to process oxide-cathode valves to an efficient level of emission using active core metals than it is using passive ones. The commonly accepted active elements in general use at present are magnesium (0.05%) and silicon (0.05%). The satisfactory production of valves using passive cores, e.g. platinum or pure nickel, is a matter of considerable technical difficulty and, except in rare cases, is not attempted today. The case for activation by chemical reduction rests on the simple reactions,

$$\begin{aligned} Mg + BaO &= MgO + Ba \\ Si + 2BaO &= SiO_2 + 2Ba \end{aligned}$$

and these taken with our opening remarks lend the appearance of powerful support to the direct reduction theory.

During the past two years a great deal of work has been carried out at Dollis Hill on the activation and oxygen de-activation of oxide-cathodes on platinum cores. From this work has come one fact of considerable interest concerning emission levels. It

appears that there is little or nothing to choose in the emission levels obtained from the 6D10 Standard Diode when fitted with cores of either platinum or active nickel. An actual example may be quoted as it illustrates the existence of spread occurring in the platinum core case just as it occurs with the nickel case. The results quoted in Table I refer to two batches of 6D10 diodes prepared and processed in apparently identical fashion. The values of total emission (TE) were measured at the usual 400 milliwatts of heater power.

Table I

Valve No.	Active Nickel Core* TE mA	Platinum Core TE mA
1	2.5	3.8
2	1.8	4.5
3	0.9	4.5
4	2.6	2.8
5	3.6	2.9
6	3.8	0.4
7	2.6	5.7
8	1.7	4.7
9	4.0	2.4
10	6.5	3.4
11	4.4	4.7
12	4.4	3.5
Mean TE	3.23	3.60
Spread of TE	0.9—6.5	0.4—5.7

* Containing 0.05 Mg and 0.05 Si.

It will be apparent that there is nothing to choose between the two groups for Mean TE level or TE Spread. Similar results to these are quoted by Herrmann and Wagener. For the benefit of those who are familiar with the processing of passive core cathodes it should be mentioned that the 6D10 pumping schedule for both types of core is the same and involves the passing of a heavy cathode current immediately after completion of the carbonate breakdown phase.

Substantial reason for doubt on the chemical reduction theory is thus made plain – the level of emission achieved by a passive core is effectively the same as that for an active core.

Clearly an anomaly has now arisen and we must attempt to resolve it before passing on to other matters. Why is it that it is so difficult to satisfactorily process a passive core valve when it appears so easy to activate a passive core cathode ? The explanation appears to be along the following lines. It should be first explained that the cathode core is coated with $(BaSr)CO_3$ before the valve is built up, and that this mixed carbonate is broken down to the (BaSr)O form under vacuum during the pumping process. The reason for preferring the carbonate to the oxide for the initial coating of the core is simple – the carbonates are non-hygroscopic and stable in damp air whereas the oxides are extremely hygroscopic. During the decomposition phase the individual crystals of $(BaSr)CO_3$ (written henceforth as $BaCO_3$ for simplicity) break down to barium oxide and gaseous carbon dioxide which is pumped away. During the breakdown a state of chaos occurs in each grain of the matrix whilst a crystal of $BaCO_3$ is losing CO_2 and then rearranging itself into the smaller and differently shaped crystal of BaO. In the sorting-out process that occurs some of the barium or oxygen atoms or ions are regarded as becoming separated from the normal Ba=O bonding and it appears to the writer that this hypothetical physico-chemical separation is the basis of the high levels of emission that are common in oxide-cathodes. Clearly two conditions are required to ensure the highest possible activation ; firstly the separated oxygen atoms or ions must be prevented from wandering around in the matrix and re-uniting with their former partners ; and secondly, additional oxygen must be prevented from entering the matrix from the atmosphere of residual gas which surrounds it. These two conditions can be realised approximately in two distinct ways. If the core metal is the usual active one, then a thin cloud of magnesium vapour will traverse the core through the intergranular spaces and will act as a sort of ' internal ' getter fixing the separated oxygen as MgO which is neutral to the matrix as a whole. Furthermore, gases invading the cathode from the outside atmosphere will be dealt with in the same manner with the overall result that the physico-chemically separated barium will remain undisturbed. The alternative method for the preservation of separated barium is more difficult and requires two special conditions ;

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a high cathode current to draw the separated negative oxygen ions quickly out of the cathode and an exceptionally high vacuum to prevent oxygen re-entering the cathode from the outside atmosphere.

In the case of the two groups of diodes recorded in Table I, the conditions of high cathode current immediately after decomposition and very high vacua have been observed, and these are thought to explain the high level of total emission of the platinum core samples. In the case of the active cores the internal gettering action of the magnesium has clearly been unnecessary and it seems to have played an insignifiicant part as a direct producer of excess barium. The role of the magnesium is thus regarded as that of a protector rather than a producer of excess barium and its technological virtue is that it enables valves to be satisfactorily processed in the presence of relatively high residual gas pressures. A direct example of the internal gettering action of magnesium will be described in the next subsection.

Two further possibilities of direct chemical reduction will be quickly considered and as quickly dismissed. During the spraying of the carbonates on to the cathode core, a binder of nitrocellulose is used to temporarily assist adhesion of carbonates to core. Processing of the valve leads first to a breakdown of the binder and then the carbonates. It is commonly assumed that carbon results from the binder decomposition and that this reacts with the CO_2 from the carbonates to form CO. The reducing agents to be considered are therefore carbon and carbon monoxide.

The writer has undertaken no experiments with the direct object of investigating the efficacy of carbon but various experiences seem to indicate that its role is at best a minor one. For example, cathodes which have been prepared by water spraying without nitrocellulose binder seem to activate in much the same way as normal cathodes. A more direct indication comes from some work on acetylene poisoning of cathodes where a thin deposit of carbon has been deposited on the granule surfaces of an activated cathode and then removed by thermal evaporation. Such treatment has not enhanced the total emission but led rather to a deterioration – possibly due to excess barium evaporation along with the carbon vapour. Herrmann and Wagener have come to similar conclusions on the small or even negligible part played by binder carbon in the direct production of excess barium.

The effect of pure dry carbon monoxide has been subject to deliberate experiment and found to have a direct and irreversible poisoning action on the activated cathode.

The general view, therefore, taken by the writer at present is that direct chemical reduction plays a small and probably negligible part in the initial activation. This must not, of course, be taken to imply that cathodes cannot be activated by reduction – such gases as methane or propane are powerful activators – but simply that reducing action is not important in the conventional process.

ELECTROLYTIC PRODUCTION OF EXCESS BARIUM

The writer has suffered much confusion over this question of electrolytic production of excess barium in the oxide-cathode. He has decided, therefore, to start by describing two cathode reactions involving the increase of emission with passage of current but which he does not regard as electrolytic phenomena.

Reaction A. Influence of Cathode Current on the Rise of Emission during Cathode Activation

Two standard diodes 6D10 identical except in the nature of the core metal were pumped down to $\angle 1\times10^{-6}$mm.Hg and the carbonates then decomposed. After completion of decomposition both cathodes were held at 1,250°K for 15 minutes with the cathode current $I_c=O$, and then for a further 12 minutes with cathode current $I_c\sim100$ mA per sq. cm. At regular intervals during the treatment snap measurements were made of low-temperature total emission. One of the cores was platinum and the other a normal active nickel. Results are set out in Fig. 2.

In the platinum case there is clearly no effective gain in emission until cathode current is passed, when the rise is spectacular in its suddenness. The active

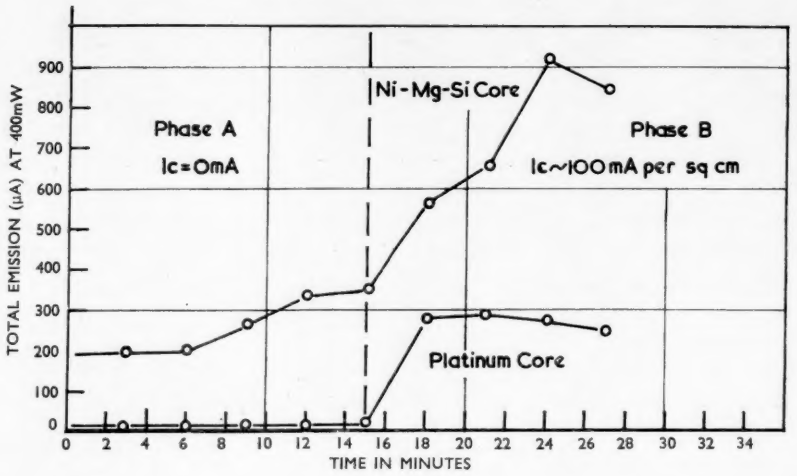

Fig. 2 The rise of total emission during initial activation.

nickel core shows a steady rise during the no-current phase due to internal gettering by magnesium but even in this case there is a marked speeding-up on applying cathode current. The low level of emission in the platinum case is thought to be due to the period of 15 minutes running before the application of the 'scavanging' current.

Reaction B. Influence of Cathode Current on the Rate of Recovery from an Oxygen De-Activation

In this case a 6D10 diode with platinum core was subjected to standard doses (1 micron-minute at 1×10^{-4}mm. Hg) of dry molecular oxygen. At the end of each dose the gas was shut off, the residual gas pumped away to $\angle 1\times10^{-6}$mm. Hg and the cathode allowed to recover. The several cycles were conducted at cathode temperatures of 1,050°K for the poisoning phase and 1,150°K for the recovery phase.

Results are shown in Fig. 3.

Recovery from the first two doses is rapid under the influence of cathode current. The third cycle starts, however, with a period of zero cathode current during which the emissive state is tested by a pulse method; negligible recovery occurs until current is again drawn when the emission rises in a manner similar to the first two cycles. It should perhaps be emphasised here that the recovery curves in Fig. 3 are space-charge limited and in the recovered condition give no indication of changes of total emission over successive cycles. This point will be brought up in the next section.

The two reactions illustrated have clearly much in common and are indeed regarded by the writer as basically similar phenomena. Now to pose the question which is the real object of introducing the two examples: Are these spectacular rises of emission under the influence of a cathode current to be regarded as electrolytic phenomena ?

The answer must lie in the definition of electrolysis

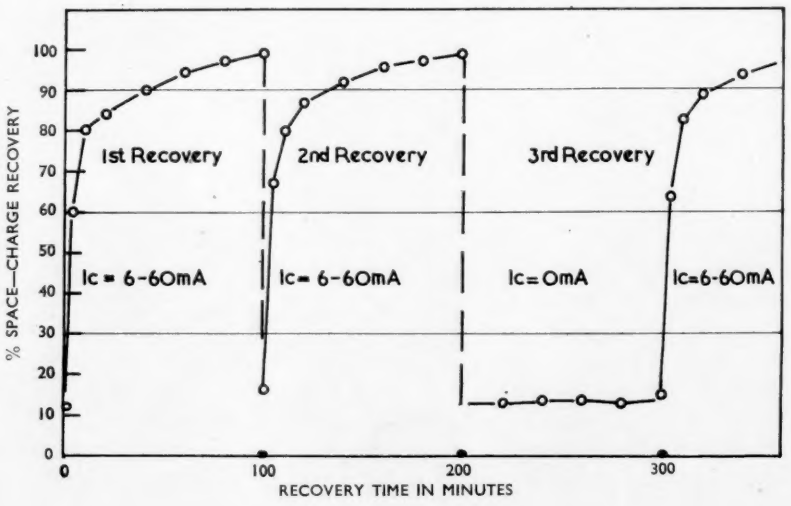

Fig 3. The influence of cathode current on the recovery rate.

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which is described as 'the decomposition of a substance by means of electricity.' The only possible substances in the cathode at the end of a poisoning phase in Fig. 3 are :

(a) The neutral matrix of BaO – or more precisely (BaSr)O,
(b) Excess barium in some elemental form,
(c) Residual oxygen gas in elemental form left absorbed in the matrix from the poisoning attack,

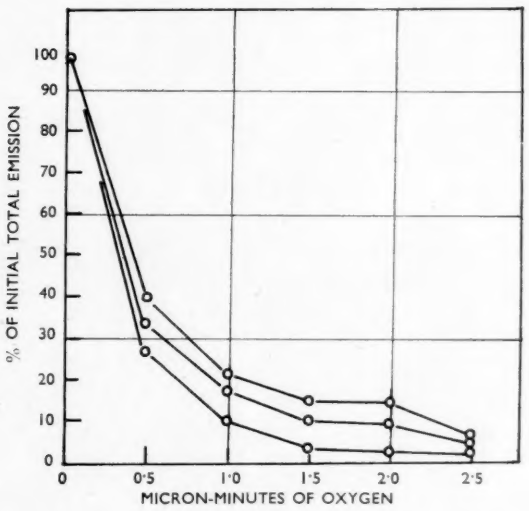

Fig. 4. The influence of molecular oxygen on the total emission.

and of these only the neutral matrix of BaO is capable of decomposition. If the recovery is electrolytic, it must therefore involve the chemical decomposition of a part of the BaO matrix. If, on the other hand, the recovery does not involve decomposition of BaO but is simply due to the physical movement of residual elemental oxygen out of the matrix, then it is not electrolytic. These are the two possibilities and both fit our reaction examples equally well.

Further experimental evidence is necessary and Fig. 4 may be useful.

A group of 6D10 diodes have been processed and their low-temperature total emission (TE) measured. The valves have then been subjected to a dose of dry molecular oxygen (0.5 micron-minute at 5×10^{-5}mm. Hg) after which the gas, has been shut off and the cathodes allowed to recover under the influence of a current until no further rise in TE is recorded. A number of similar cycles make up the results of Fig. 4. These results are interesting as they show that each successive dose of oxygen causes a permanent lowering of the level of total emission and presumably indicates a corresponding permanent decrease in the excess barium content of the matrix. Recovery from an oxygen poisoning is therefore only partial and cannot be made complete with any reasonable expenditure of cathode coulombs. If, for example, a cathode recovers to a maximum but depressed level of emission in 20 hours, then there is no observable further increase in the next 100 hours of operation. This basic irreversibility is extremely difficult to reconcile with electrolytic action. If the early part of the recovery is indeed due to decomposition of BaO on a faradaic basis, then, why does it stop short of complete recovery when such an overwhelming adequacy of BaO remains awaiting dissociation ? The writer finds no answer to this question and, partly for this reason and partly because the alternative hypothesis has proved much more attractive experimentally, he feels compelled to reject electrolytic action as a means of rapid current-assisted cathode activation or re-activation.

Before proceeding to an example of what appears to be a true electrolytic activation, the writer is anxious to give brief attention to the supposed nature of the recovery of a cathode from a bout of molecular oxygen poisoning. This recovery phenomenon is thought to be of prime importance as it is regarded as an action closely analogous to that of initial activation. The sequence of events during a poisoning-reactivation cycle is assumed to take the following course. The excess barium in the matrix is regarded as uniformly distributed throughout each BaO crystal having a small proportion (say 0.01%) lying on the crystal surface and the remainder embedded in some way within the crystal lattice. The excess barium atoms are capable of movement under concentration gradients and the rate of movement is a function of

temperature. During a poisoning attack by molecular oxygen the BaO crystal surfaces become covered to a greater or lesser extent with a layer of adsorbed oxygen which promptly attacks the surface-lying excess barium converting it permanently into neutral BaO. The crystal surfaces become denuded of excess barium and the emission therefore falls to some low value. A concentration gradient has now been set up between surface and interior of the crystal and a diffusion flow of excess barium is started. The depressed value of the emission current is thus a function of the pressure of the poisoning gas which determines the concentration of surface adsorbed oxygen, and on the cathode temperature which settles the rate, at which the reserves of excess barium flow to the surface to replace those lost by permanent chemical combination. Such is the poisoning action which implies a steady and permanent destruction of excess barium. The re-activation is simplicity itself - the excess gas is pumped away, the adsorbed oxygen on the crystal surfaces is detached under the influence of a current flow, and the cathode gradually re-establishes itself at a new and lower level of total emission appropriate to that quantity of excess barium which has escaped destruction by oxygen. The re-activation is thus regarded as purely one of mechanical separation assisted by current flow (and internal magnesium gettering in the case of active cores) followed by re-establishment of a uniform but depressed excess barium concentration throughout each BaO crystal.

The hypothesis seems to be a fruitful one as it leads immediately to the following three conclusions.

(a) Every oxygen attack leads to a permanent lowering of the excess barium concentration within the BaO crystals.*

(b) At constant cathode temperature the rate of excess barium destruction will be proportional to the pressure of the poisoning attack up to the stage at which each excess barium atom is destroyed as soon as it has diffused up to the crystal surface. Thereafter, the destruction rate is independent of the pressure of the poisoning attack.

(c) At constant pressure of the poisoning attack the rate of destruction of excess barium is a function of the rate at which excess barium diffuses to the crystal surface, i.e. the rate of the destruction increases with the rise of cathode temperature.

Work at Dollis Hill on molecular-oxygen poisoning of platinum core oxide-cathodes has supported each of these points by experiment and results will be published in due course. The postulate (b) is interesting as it seems to indicate that the invading oxygen gas waits on the crystal surface for the arrival of the diffusing barium without itself entering the crystal.

Now at last the case for electrolytic production of excess barium will be considered. Molten polar crystals are usually good electrolytic conductors and this conductivity survives the transition to the solid state but falls rapidly with decreasing temperature. The electrolytic action may take one of three forms depending on the nature of the solid electrolyte – one ion may move, or the other, or both. According to Jost[2] only the negative ion is mobile in $BaCl_2$, $BaBr_2$ and BaF_2. In the light of this information we are reasonable in assuming that electrolysis occurs in the heated oxide-cathode and involves the steady increase of excess barium concentration by removal of oxygen on a Faraday basis. The only point of contention appears to be the rate at which the action proceeds per milliamp of cathode current at the normal running temperature of 1,050°K.

Unfortunately positive information concerning electrolytic generation of barium is meagre and the writer has fallen back on such information as he can find at Dollis Hill. Some indications can be gathered from experience of the loss of matrix in valves which have passed very large amounts of electricity. In one such case a valve type passed some 850 ampere-hours in 20 years and still retained much of the matrix on its cathodes at the end of the run. Some experiment by Wagener[3] arranged for a group of

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* In some earlier experiments (*Nature*, 164, (1949) 589), an apparent reversibility to oxygen poisoning was demonstrated. It is now known that in this previous work the magnitude of the oxygen attack used was too small to ensure detection of its effects.

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close-spaced diodes to pass some 2,000 ampere-hours in 8,000 hours. Comparison of the cathodes at the end of the run with control specimens showed no obvious differences in matrix bulk. These essentially qualitative observations show at least that electrolytic transport at normal cathode temperatures must be an extremely slow process. Perhaps the best and certainly the most direct way of demonstrating electrolytic production of barium is by observing the change of total emission with time in platinum core cathodes carrying current. Results shown in Fig. 5 refer to 6D9 diodes running at a nominal 1,050°K and the overall rise in total emission is regarded as due to an increase in excess barium concentration by electrolytic action. The diodes were fitted with barium getters and it is assumed that the increase in excess barium content is matched by an equivalent absorption of oxygen by the getter. If the cathodes (3 milligrams of matrix) contain 3 micrograms of excess barium at the beginning of life then the characteristics of Fig. 5 indicate that the loss of matrix weight by electrolytic transport is not likely to exceed 10% in 20 years. Clearly the electrolytic action is a slow one and the writer disregards it as a practical means of initially activating cathodes to normal levels of emission.

THERMODYNAMIC ACTIVATION

The theory of lattice defects in ionic crystals developed by Frenkel, Wagener, Schottky and Jost has been applied to the conduction and emission phenomena of oxide cathodes with considerable success. It appears that a crystal of BaO requires the presence of lattice defects or vacancies to achieve thermodynamic equilibrium at any particular temperature. Assuming that defects of both kinds (oxygen and barium) are produced it only requires some mechanism such as current flow to remove oxygen in order to leave the cathode stocked with an excess of barium. This action will not, of course, be electrolytic as the initial separation from the normal chemical bond results only from a postulated thermodynamic necessity. The concentration of excess barium that is likely to arise in this way is an exponential function of temperature and follows some such law as,

$$N = Ae^{-\frac{W}{kT}}$$

For a while the writer accepted the thermodynamic mechanism as a satisfactory medium for explaining initial activation but has gradually developed doubts as to its adequacy. The reason for this scepticism is simple and can be explained in a few words. Once a platinum core diode of the 6D10 type has been fully activated, it has never been found possible in the writer's laboratory to increase the level of total emission by thermal means either in the presence of a current flow or otherwise. In fact, once activation has been completed an increase in cathode temperature has always resulted either in no change of low temperature emission level or an actual decrease which is assumed due to evaporation of excess barium. This consistent failure to increase the emission level of an activated cathode is matched by a corresponding inability to complete re-activation of an oxygen poisoned cathode to its former level by thermal means or by current flow. These facts are difficult to reconcile with an exponential relationship between barium concentration and temperature which seems

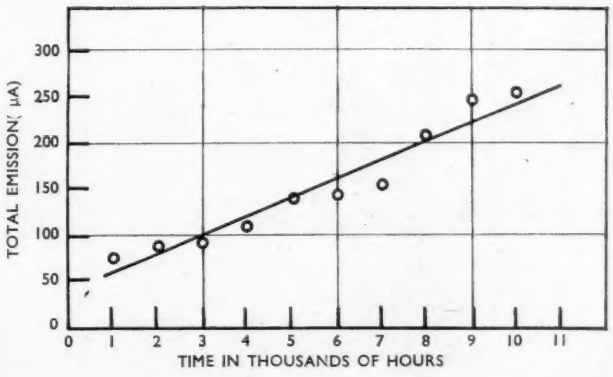

Fig. 5. The rise of total emission (TE) during the life of platinum core diodes (6D9). The graph represents an average of 6 samples.

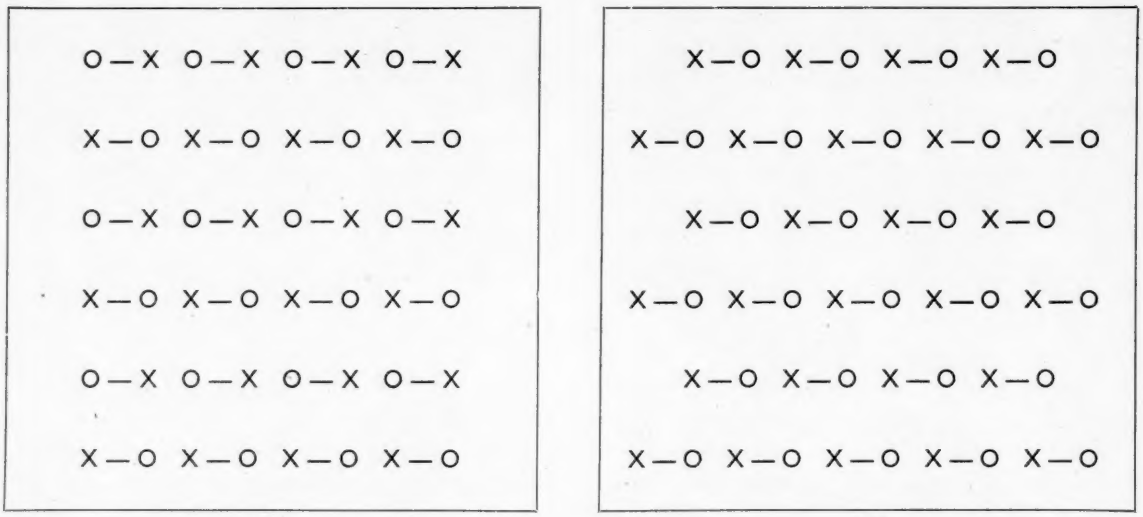

Fig. 6. Transition phenomena assumed to occur between the times of carbonate breakdown and growth of oxide crystals. (*a*) Shows a plane through a growing BaO crystal where one BaO molecules lies directly above a previous molecule : growth by columning. (*b*) Shows a plane through a crystal where a BaO molecules places itself across two previous molecules: growth by bridging. Note: The symbol O-X represents BaO molecule in isolation. The individual bonding is retained in the diagram to illustrate the two different possibilities of association of the molecule with the growing crystal.

implicit in the thermodynamic theory. The writer has given some thought and experimental effort to this anomaly and feels at present that a way out of the difficulty may be found by assuming that there are two natural but widely differing levels of emission in the oxide-cathode. Immediately after initial activation the excess barium concentration is of the order of 0.25% of the matrix weight and it is assumed that this has been produced by two separate and distinct processes. The first process, responsible for about 1 part of the excess barium, is thermodynamic and follows the exponential law with regard to temperature ; the second is a physico-chemical one and is responsible for the production of about 9999 parts of the excess barium. This physico-chemical process is regarded as a 'one-time' reaction which occurs during the build-up of the BaO crystals from the decomposing carbonate and can never thereafter be repeated. Raising the cathode temperature results in a relatively insignificant increase in excess barium by thermodynamic action and, coincidentally, a relatively large loss of barium by evaporation from stocks initially installed by the physico-chemical action. The overall effect of a temperature rise is therefore a lowering of total emission level.

This picture is essentially speculative but it has been built up from experiments which may be summarised in the following manner. During work on the oxygen de-activation of platinum core diodes it has been found extraordinarily difficult to reduce the emission level below a certain value. After permanent reduction of total emission by a factor of the order of 10^4 it seems that any further reduction by oxygen is of a temporary nature and is reversed when the poisoning action is discontinued. For example, a batch of platinum core oxide-cathode diodes were maintained in an atmosphere of 1.0×10^{-5}mm. Hg of oxygen for 200 hours : at intervals of 24 hours during this treatment the gas pressure was reduced to $\angle 1.0\times10^{-8}$mm. Hg and the cathodes allowed to recover to a maximum value of emission. The maximum value over the last 100 hours of the poisoning action was found to be substantially constant. This 'run-down' state of the emission

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level seems therefore to be a natural condition of the cathode to which it returns each time after an oxygen attack. Further work on the 'run-down' state has shown that the approximate constancy of emission level, to which the cathode returns after each attack is dependent on constancy of cathode temperature during the re-activation periods. If the temperature is raised during a re-activation phase, then the maximum emission level achieved is also raised. The level achieved at 1,250°K – a common activating temperature for commercial cathodes – is still far below that usually achieved in initial activation. As might be expected the periods required for recovery fall rapidly with increasing cathode temperature.

From such experiments as these the writer is at present inclined to think that the 'run-down' emission levels may be a thermodynamic phenomenon occurring several powers of ten below the initial activation level. Work on the problem continues but it is a laborious one to handle from the vacuum point of view and it will be some time before firm opinions begin to develop. On one point, however, the writer is becoming convinced – that the initial activation level is not brought about by a straight thermodynamic action in a formed crystal lattice of (BaSr)O.

FINALE

Discussion so far has been almost entirely destructive in the sense that each of the three major hypotheses of initial activation has been made the subject of doubt. In their place has been introduced a wholly vague conception of some physico-chemical reaction which is assumed to occur as a transition phenomenon functioning between the times of carbonate breakdown and growth of oxide crystals. The manner of such a reaction is of course anyone's guess and the article may excusably wind itself up with one by the writer. This idea is disarmingly simple and involves the random way in which a BaO molecule leaves the chaotic condition of carbonate breakdown and seeks to associate itself with the surface of a growing BaO crystal. As the molecule approaches the surface it has two choices of action – it may lie directly above a previous molecule (columning) or it may lie across two previous molecules (bridging). Fig. (6a) shows a plane through a crystal built up wholly by columning and Fig. (6b) one built up by bridging. Two cubic crystals built up in these two ways will be identical in all respects, except that in the bridging case two opposite faces of the cube will be covered respectively with an absorbed layer of oxygen and an absorbed layer of barium respectively. The essential dissociation has thus been effected and the likely degree of activation can be calculated. Assuming a cubic shape of the grown crystal, equal probability of columning or bridging, and complete removal of the separated oxygen, then the activation is given by :

$$\text{Per cent Excess Barium} = \frac{25}{N^2}$$

where N is the number of molecules which go to make up one crystal edge. According to this view the degree of activation can be increased indefinitely by decreasing the size of the crystal. Assuming that crystal size in normal cathodes is around a micron, then the calculated activation is about 0.01% – the values quoted by Herrmann and Wagener lie between 0.01% and 0.25%.

References

1 G. HERRMANN and S. WAGENER, *The Oxide-Coated Cathode* (Chapman & Hall Ltd. London, 1951).

2 W. JOST, *Diffusion und Chemische Reaktion in Festen Stoffen* (Steinkopff, Dresden, 1937) 80.

3 S. WAGENER, *Nature*, **164**, (1949), 357.

CONFERENCE REPORTS

SYMPOSIUM ON FREEZING AND DRYING

DURING JUNE 29th and 30th 1951, the Institute of Biology held a symposium on ' Freezing and Drying ' in London.

As a laboratory procedure 'freeze drying'—or, more appropriately, ' drying by sublimation '—has been known for many years, the first clearly recorded use for the preservation of biological materials being described by Shackell in 1909. It was the last war, however, with its huge demands for dried plasma and for penicillin, which gave the greatest impetus to the technique.

Despite the present widespread use of this method of preservation it must be admitted that knowledge of the technique and criteria for satisfactory drying often appears to be rudimentary. There can be no doubt that this symposium served a useful purpose in the pooling of ideas and in the extension of this knowledge.

The symposium's first session, with Dr. R. I. N. Greaves in the chair, was devoted to a discussion of the industrial application of freeze drying with special reference to antibiotics and food-stuffs. Papers in the second session, with Professor Sir Alexander Fleming, F.R.S., in the chair, reported the preservation of mammalian, avian and plant cells at low temperatures. The third session, with Dr. C. H. Andrewes, F.R.S., in the chair, dealt with the freeze drying of bacteria and viruses, and Professor J. F. Danielli was in the chair for the last session on the subject of tissue fixation by freezing and drying.

Each session was attended by 300-400 people including a large number from overseas and it was found possible to record all the discussions on the papers for inclusion in the full report of the symposium which the Institute of Biology will publish in May, 1952.*

In addition to the formal business of the meetings, W. Edwards & Co. (London) Ltd., arranged a reception at their Sydenham factory which was attended by a large proportion of the members. A wide selection of equipment for freeze drying and ancillary purposes was demonstrated and opportunities were given for visitors to discuss these with the designers.

The following are summaries of various papers presented at the symposium.

*The report is published under the title ' Freezing and Drying ' and can be obtained from Mr. D. J. B. Copp, General Secretary, Institute of Biology, Tavistock House South, Tavistock Square, London, W.C.1, England (Tel. No. Euston 1040). Price 15s. 0d.

Some Applications of Freezing and Drying in Cytochemistry.

Presented by : *L. G. E. Bell.*

The tissues are frozen in liquid propane at —187°C. rather than in *iso*pentane. When the tissue temperature is maintained at —40°C. the drying cycle occupies two days. The dry tissues are then embedded in degassed wax at 54°C., cut and dry mounted. The chief advantage of freeze drying does not lie in the retention of structural detail but in that cell constituents are unaltered in position and in composition. In chemical fixation, diffusion of cellular material gives rise to serious artefacts and the cytochemical results are thus only acceptable, if it can be shown that the constituent tested for is neither removed nor translocated.

Freeze Drying of Tissues for Cell Analysis by Light and Electron Microscopy.

Presented by : *F. Sjostrand.*

The freeze drying of tissues for cytological investigation has been evaluated on the principles of the maintenance of the structure of the cells and the preservation of some chemical components of the cell.

Dark ground microscopy is most useful in studying vacuolisation of ground cytoplasm and nuclei as a result of ice-crystal formation and the degree of preservation of the orientation of anisodiametric submicroscopic elements has been investigated by analysis in polarized light.

It has been found that freezing should be as rapid as possible preferably of sections only a few tenths of a millimetre thick. Moreover, within a few minutes of the death of the host, changes in the structure of the mitochondria of kidney tubular cells become evident and speed of work is important.

Chemical composition of frozen-dried tissue has been investigated by fluorescence microspectrography and results are shown for riboflavin in the proximal tubules of guinea-pig kidney.

For electron microscopy a new technique for the

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preparation of ultra-thin sections has been developed. The problem of artefacts is more pressing in the case of electron microscopy and much systematic work remains to be done.

Some Recent Experiments in Fixation by Freezing and Drying. Presented by : *I. Gersh.*

The use of polyethylene glycols as embedding media has now made it possible to prepare frozen and dried materials for the study of their lipids and an extensive investigation of plasmalogen has been made.

The calculations of Dr. J. L. Stephenson have shown that, providing that the design of the vacuum system of the tissue freeze-dryer is adequate, the limiting factor in the speed of drying lies in the tissue itself. Thus, when the apparatus was designed as essentially a molecular still the resistance to water-vapour diffusion at 10^{-4}mm. Hg caused by a shell of dry tissue 1 mm. thick was about 10^4 times that of the rest of the apparatus ; when the vacuum line was lengthened and narrowed to resemble the usual apparatus the corresponding factor was 45.

Effects of Freezing and Drying Compared With Common Fixation Procedures. Presented by : *B. Sylvén.*

Qualitative tests indicate that proteins, nucleotides, lipids, carbohydrates and polysaccharides are lost from tissues subjected to the common fixation procedures. The total loss of mass during complete histological processing of parenchymatous organs, such as the liver, is of the order of 40% to 60% of the original tissue mass. The limitations of the freezing and drying technique are not to be found in these procedures *per se*. However, the subsequent treatment of the tissue is most important and has been insufficiently investigated. It is possible that the almost complete preservation of cytoplasmic constituents by freeze drying is a result of denaturation, and thus increased insolubility of the tissue proteins.

The Influence of the Suspending Fluid on the Survival of Bacteria after Drying. Presented by : *R. M. Fry.*

All the suspending media tested appeared to be improved by the addition of 5% to 10% glucose or lactose which probably prevents the bacteria, cultures from being over-dried. For optimum results a protective colloid was also necessary. Broth was quite suitable for ' D 201 H ' strain paracolon bacillus, which is a fairly resistant organism and albumin, haemoglobin, serum and milk all gave good results. For delicate organisms such as *N. gonorrhoeae*, *Vibrio cholerae*, *H. influenzae* and *H. pertussis*, ' *Mist. desiccans* ' has been developed, containing 1 part nutrient broth and 3 parts serum with a final concentration of 7.5% glucose. In such a medium *Vib. cholerae* gave 1% survival after storage for 3 years.

Some of the Factors Affecting the Viability of Bacterial Cultures Subjected to the Freeze Drying Process. Presented by : *H. Proom.*

More than 2,500 bacterial strains are being preserved in a desiccated state at the Wellcome Research Laboratories.

Spore-forming bacteria and Gram-positive cocci may be dried by almost any method and, if they are kept dry, will survive for very long periods. For less resistant organisms the composition of the suspending medium is of paramount importance, and protective colloids and reducing substances should be present. In the freeze drying procedure the protective colloid must reduce the immediate death following freezing. It is important that the temperature of the suspensions during drying should not rise above —30°C.

So far as loss of viability during storage is concerned two factors operate at any one temperature, the presence or absence of water and oxygen. For optimum survival secondary drying is necessary, and the dried cultures should be sealed in dried nitrogen or *in vacuo* and stored at the lowest practicable temperature.

Infection and Drying Techniques. Presented by : *S. T. Cowan.*

In large-scale drying of bacterial cultures it is impossible to cap each tube separately. Prevention of contamination by airborne bacteria was accomplished by fitting a multiple-layer cheesecloth cover over the whole head of the centrifugal freeze dryer.

When it is desirable to dry different cultures in the same vacuum chamber at the same time, the possibility of cross-infection must be investigated. Experiments with a combination of sterile tubes, a

salmonella and a streptococcus showed that 10% of the tubes were cross-contaminated. Fluorescent tracers showed that ' spills ' were greater from water than from serum. Segmentation of the centrifuge head, each segment with a separate gauze cover, overcame the difficulty.

There is a risk of human infection by aerial contamination during the filling of ampoules for freeze drying. There is a further risk in the use of weak or cracked ampoules and in careless technique in opening ampoules of dry material.

The Freeze Drying of Living Virus Vaccines for Veterinary Use. Presented by : *R. Daubney.*

Rinderpest vaccine, derived from infected goat spleen, may be frozen-dried and issued at very low cost. Such material has already given life-long protection to millions of native cattle of such low economic value that they did not warrant the expense of immunisation by older methods. Vaccines are also available for Newcastle disease and fowl plague.

Recently chick-embryo-adapted rinderpest vaccine has been produced and dried in a new plant designed to handle 10 litres (100,000 doses) at a time. The author discusses in detail factors in the drying procedure, such as the preparation of the material, rate of application of heat to the drying product, residual moisture and temperature of storage, which determine the overall loss of viability.

A Difficulty in the Quantitative Freeze Drying of Certain Materials. Presented by : *Wilson Smith.*

Some materials, such as dilute solutions of salts, cannot be freeze-dried quantitatively by the usual procedure in a centrifugal freeze dryer. Within a short while of snap-freezing, ' explosions ' occur in the ampoules and particles of the frozen solution are ejected. Addition of very small quantities of protein to the solution appears to reduce this loss considerably. It is difficult however to think of a simple way of overcoming this hazard with standard ampoules and phials and without contaminating the product.

The Preservation of Vaccinia Virus by Freeze Drying. Presented by : *L. H. Collier.*

A study of two of the variables involved namely (a) the nature of the suspending material in which the virus is to be dried and (b) variation of the drying procedure. The following conclusions were obtained.

(1) When a suitable protective medium is employed, frozen-dried vaccinia virus may be stored at 22°C. (or at 37°C.).
(2) Suitable protective media are (i) the extraneous matter present in vaccinial sheep pulp and (ii) digest broth, 1% (or 5%) peptone and 10% normal horse serum.
(3) Drying technique was important in determining subsequent virus survival but the precise optimum conditions have not yet been defined.
(4) Dried virus should be stored *in vacuo* rather than in dry nitrogen.

Contributions on Problems Encountered in Drying Viruses, Vaccines and Antisera by Sublimation. Presented by : *I. A. Galloway.*

Early experiments had shown that freeze drying of foot-and-mouth disease vaccines lead to a loss of immunising potency of about 50% ; a loss which was not a result of the freezing.

Even after long periods of storage, frozen-dried complement can be reconstituted and used satisfactorily without re-titration.

The potencies of anti-sera to different types and and strains of foot-and-mouth disease virus are maintained when they are stored in the liquid state at 4°C. or frozen, but freeze drying gives a product with increased stability, especially with regard to preservation at higher temperatures and ease of transport.

Freeze drying of anti-sera may have no effect on the antibody titre but may increase the anti-complementary properties of some anti-sera or lead to their development in anti-sera not originally exhibiting them.

The Viability of Mammalian Skin after Freezing, Thawing and Freeze Drying. Presented by : *R. E. Billingham & P. B. Medawar.*

The malpighian cells of the epidermis of rabbits' ear skin will survive either very rapid or very slow freezing. These cells, in grafts soaked in Ringer's solution, do not, however, survive slow thawing but will do so if the grafts are soaked, before freezing, in Ringer's solution containing 15% glycerol. Epidermal

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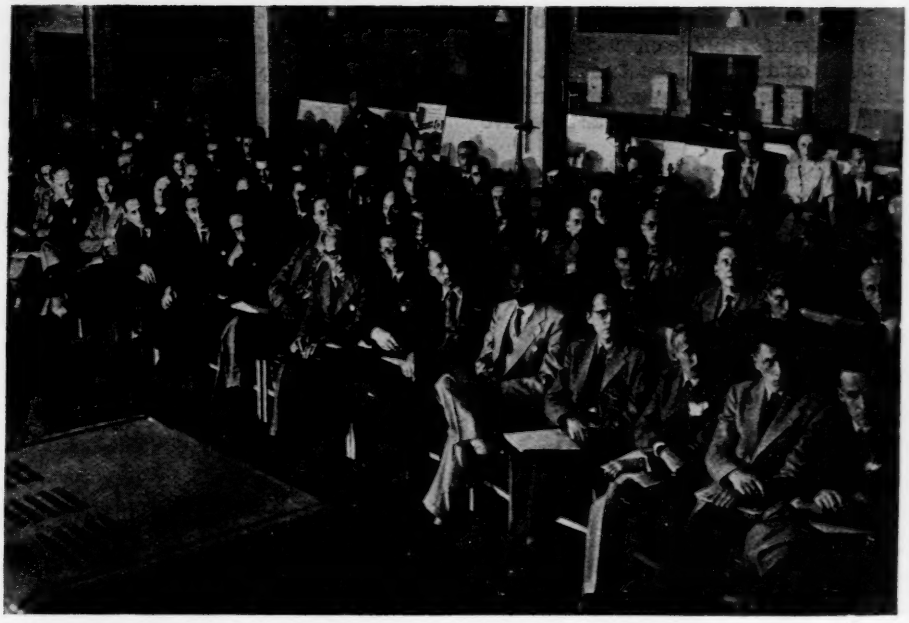

Symposium on Freezing and Drying. Visitors to the exhibition listening to the address of welcome at the reception. In the front row, third from the left: Dr. R. J. C. Harris the author of this article.

melanoblasts in skin soaked in Ringer's solution alone withstand neither fast freezing nor slow thawing. Pre-treatment as above, however, enables them to withstand the fast freezing but not subsequent slow thawing. Suspensions of isolated epidermal cells will withstand slow freezing in the Ringer—15% glycerol but not in Ringer alone.

On all grounds upon which comparison can be made *slow* freezing of skin is preferable to fast.

Skin soaked in Ringer, or in Ringer containing different concentrations of glycerol could not be dehydrated below a final water content of about 25%.

Freeze Drying of Tumour Tissues. Presented by: *R. D. Passey & L. Dmochowski.*

The tumour tissues (spontaneous and induced sarcomata and carcinomata of various inbred strains of mice) were minced finely, suspended in glucose solution, frozen in crushed, solid carbon dioxide and stored at —79°C. Specimens to be freeze-dried were then held at —25°C. to —30°C. for the period of drying (1 to 3½ hours in the apparatus described). Length of storage at —79°C. was found to have no influence on the tumour-producing potency of the suspension.

Histological investigation of cells in the various suspensions before and after freezing-thawing, after freeze-drying and before and after reconstitution led to the conclusion that at least some of the cells appeared to survive both freezing and freeze drying and this was confirmed by growing such cells *in vitro*. The water contents of the frozen-dried products have not been determined but it does not seem likely that desiccation has been complete in cases in which cells have been shown to survive.

Survival of Cells, Tissues and Organisms after Ultra-Rapid Freezing. Presented by: *B. J. Luyet.*

Details are given of the freezing and thawing of muscle fibres, chick embryo amniotic muscle, chick embryo heart, red blood cells, 'vinegar eels,' frog spermatozoa, moss leaves and the epidermal cells of some plants. The results are held to confirm in general the predictions of the theory of vitrification by very rapid cooling especially with regard to (a)

the necessity for a high freezing 'velocity,' (b) the need for rapid re-warming, (c) the effect of an initial reduction in water content, (d) the existence of a limited range of 'dangerous' temperatures and (e) the innocuity of very low temperatures once they have been obtained.

Techniques are described for obtaining high cooling 'velocities' and it appears that *iso*pentane at —150°C. gives a rate of cooling which is five times as fast as that obtained with liquid nitrogen at —195°C. An apparatus for the determination of cooling 'velocities' enables 'speeds' of up to 150,000°C. per second to be recorded.

Preservation of Spermatozoa, Red Blood-Cells and Endocrine Tissue at Low Temperatures. Presented by : *A. S. Parkes.*

Spermatozoa. Fowl semen, diluted with Ringer's solution containing 40% glycerol and frozen for twenty minutes at —79°C., resumed full motility on thawing and could be stored for as long as five months at —79°C. Slow cooling (1°C./ minute from 0°C. to —79°C.) of suspensions of bull or goat sperm containing 20% glycerol was compatible with the survival of a large proportion. Slow removal by dialysis of the glycerol in the thawed fowl semen suspension gave sperm with normal fertilising power. Similar treatment for the mammalian sperms was not so successful.

Red Blood-cells. Whole rabbit or guinea-pig blood diluted to a final concentration of 10% to 15% glycerol and frozen to —79°C. could be recovered after thawing even after storage for three months. The glycerol could be removed by dialysis and it appears likely that long-term storage of human blood (especially rare types) will be possible under such conditions.

Endocrine Tissue. The cumulus cells of rabbit ova survived and grew in tissue culture after slow or rapid freezing in solid carbon-dioxide or in liquid air.

Fragmented (but not whole) adult rat ovaries suspended in glycerol-saline showed good resistance to freezing. Grafts of thawed tissue back into the donor animal showed that these were endocrinologically active but were devoid of ova.

Large Scale Freeze Drying as Currently Carried out in the Western Hemisphere. Presented by : *E. W. Flosdorf.*

A general survey of the methods current in the United States for large-scale freeze drying.

Procedures discussed include pre-freezing and freezing *in situ ;* for the supply of heat to the drying product by means of circulating hot water, by direct electrical heating or by infra-red irradiation. Systems for the maintenance of vacuum in the drying chambers include oil pumps in conjunction with mechanically-refrigerated condensers or multi-stage steam ejector pumps which pump the water-vapour directly. A large variety of frozen-dried products are now available commercially in the Western Hemisphere including human blood products such as immune serum globulins ; enzymes, antitoxins, antibiotics, hormones, veterinary vaccines, laboratory biological reagents such as complement, and food products such as coffee, pineapple juice, orange juice and raw, ground beef.

Freeze Drying of Antibiotics on a Commercial Scale. Presented by : *J. H. Singer.*

A freeze drying plant for drying small quantities of penicillin solution in large numbers of individual vials is described and illustrated. Eight separate drying units had each a direct expansion refrigerator (which maintained the condensing coil at —50°C. under full load) and were connected in two banks of four units to manifolds evacuated by two-stage mechanical pumps. The bottles were warmed up to about 40°C. at the end of the run, reducing the final moisture content to well below 1.0% without secondary drying. With four drying runs in twenty-four hours, each unit produced a daily output of 10,000 vials. The original fabric sterility lids for the trays of vials were replaced by plain metal lids which remained in place during freezing and loading into the drying units, but which could be raised and lowered by an external hydraulic control.

A bulk drying plant described and illustrated for freeze drying streptomycin calcium chloride complex and dihydro-streptomycin sulphate was capable of drying more than 300 litres of solution in a 48-hour cycle. Solution in the trays was frozen *in situ* by allowing refrigerant to flow through the shelf coils.

Freeze Drying of Foodstuffs. Presented by : *R. Gane.*

The author describes in detail the design and operation of a freeze drying unit which (a) gave very rapid rates of evaporative freezing, (b) was

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symmetrical enabling various samples with different pre-drying treatments to be dried at the same time under comparable conditions, (c) was able to record electrically changes in the weight of a sample during drying and (d) had thermo-couples in the drying masses.

Results are shown for the drying of sliced apple, apple juice, tea (which, however, loses volatile odorous substances), minced beef and fish fillets. Liquid egg is difficult to freeze and loses CO_2, giving an alkaline product after reconstitution. Powders from sugar-containing liquids caked or even partially liquefied on storage—probably due to subsequent crystallisation of initially-amorphous sugars.

Reconstitution of meat and fish tended to be difficult if the pieces were more than 1 cm. thick. Vegetables, such as peas, with an impermeable skin could not be frozen by evaporation and pre-freezing and grinding was essential.

Aspects of the Design of Freeze Drying Apparatus. Presented by : *A. S. D. Barrett & L. G. Beckett.*

A detailed discussion of some of the factors which influence the design of laboratory freeze-dryers for general use.

The eutectic point of the material to be dried determines the design of the vacuum system since the lower the eutectic point the greater are the volumes of water-vapour which have to be removed from the system.

Vacuum pump requirements are determined by the speed of the pump and the resistance of the vacuum line. For most laboratory freeze drying a safe pressure of 0.5 mm. Hg is quite satisfactory. Mechanical vacuum pumps are usually employed in conjunction with a refrigerated condenser and it is important that, under these conditions, the lowest pressure attainable with the pump should be not less than the saturation vapour pressure of ice at the temperature of the condenser, otherwise the pump will tend to strip the ice from the condenser.

For all-glass freeze-dryers the usual method of refrigerating the condenser is with a solid carbon dioxide-alcohol bath. Each gramme of ice condensed will require a minimum of 6 grammes of solid carbon dioxide. Larger equipment usually employs mechanically-refrigerated condensers.

Chemical desiccants (and phosphoric oxide is the best) may be used for work on a small scale but a minimum of 3 grammes of desiccant is required for each gramme of water removed.

The most generally effective pressure gauge for freeze drying systems is the Pirani gauge. The pressure range covered (1 micron to 10,000 microns Hg) is satisfactory and the instrument is robust, gives continuous direct indication of pressure and may be connected to remote indicators and recorders.

R. J. C. HARRIS.

LETTERS TO THE EDITOR

Effects of the Evaporation Technique on Optical Glass

Sommaire

LE PROFESSEUR TOLANSKY fit remarquer dans un article précédemment publié dans ce journal, qu'il avait observé que des surfaces optiquement planes en verre ou quartz étaient parfois détériorées pendant la déposition d'un film métallique par projection cathodique. L'auteur signale une observation semblable qu'il fit dans des cas où le procédé d'évaporation normal pour l'argentage de surfaces optiques était utilisé. Dans un cas une convexité de 1 à 2λ s'était formée dans le verre.

IT IS OF INTEREST to read that Professor Tolansky[1] has found that permanent damage to glass or quartz optical flats sometimes occurs during cathodic sputtering of a metallic film on to the surface.

Some time ago a similar effect was observed at this laboratory when using the normal evaporation technique. A silver film was evaporated on to the surface of a 6-inch diameter optical flat. The surface was cleaned initially in the usual manner with soapy water, nitric acid and hydrogen peroxide. The surface was finally subjected to ionic bombardment at 3,300 volts for 6 minutes. The silver was evaporated from a molybdenum boat at a current rating of 55 amperes in a chamber evacuated to 10^{-5}mm. Hg by oil diffusion pumping.

This silvered optical flat, which was originally flat to λ/20 (λ=5,000 Å) was examined using a master optical flat of known flatness. The interferograms obtained are shown in Plate I Figures 1 and 2. Figure 2 is just a different arrangement of the fringes to that in Figure 1 showing very vividly the nature and location of the 'hill' on the surface. The hill is clearly between λ and 2λ high.

The silvered flat was then thoroughly cleaned of all the visible coating of silver and resilvered by evaporation. The hill remained and continuous soaking in nitric acid for hours did not remove it. The flat was examined in white light by eye at oblique incidence and a 'burnt' region is clearly visible extending over an area of about $\frac{3}{4}$ in. Figure 3 of Plate I shows a picture of this area taken in green light. The inner part of the area is red to yellow changing to blue-violet in colour at the outer edge. There was some slight evidence of a small distortion effect on the non-silvered side of the flat.

It would appear that silver or molybdenum or impurities in the evaporation chamber have been

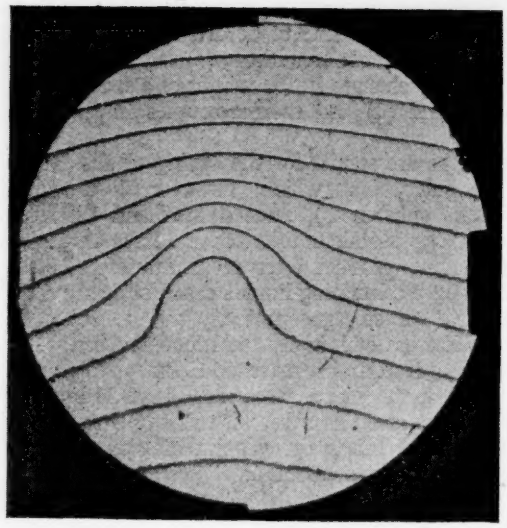
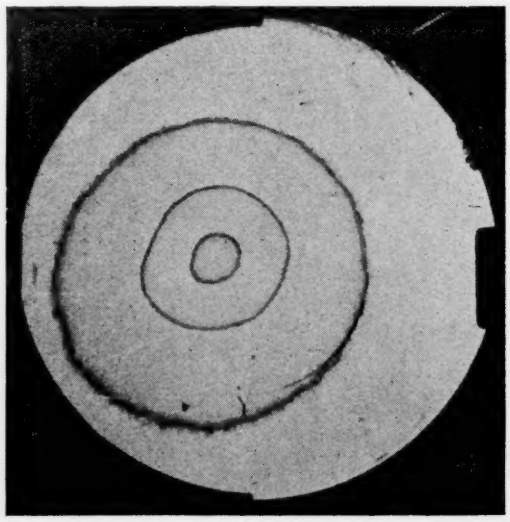


Fig. 1.

Fig. 2.



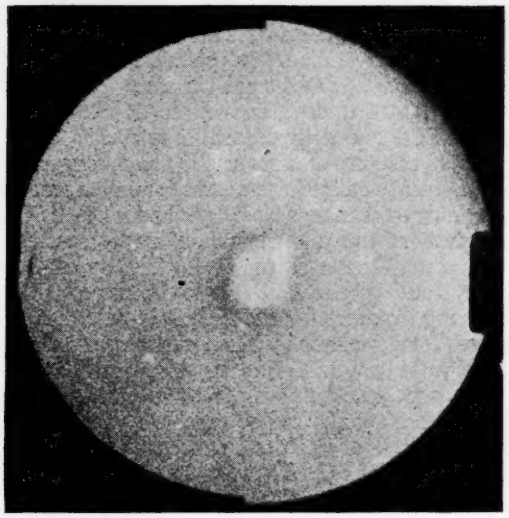


Fig. 3.

driven into the surface from a sudden but unnoticed burst of ions or particles during the bombardment or evaporation. It is proposed to remove the top surface layer of the flat chemically and have this examined by micro-analysis.

It is worth noting that a 3-inch diameter flat made of optical glass was affected similarly a short time later. Further experiments were done with a test piece of optical glass using deliberately excessive bombardment rates and heating currents but the effect could not be repeated. Examination of the glass with polarised light during bombardment and evaporation showed no evidence of strain taking place in the glass. Further investigation of the exact nature of the material deposited on a surface by evaporation seems desirable when effects such as those described above can occur.

C. F. Bruce.

National Standards Laboratory,
University Grounds,
Sydney,
Australia.

1st October, 1951.

[1] Tolansky, S., *Vacuum* 1, (1951), 75.

BOOK REVIEWS

Industrial High Vacuum. By J. R. Davy. (Sir Isaac Pitman & Sons Ltd., London, 1951.) Pp. 243. Price £1 5s. 0d.

WHEN WRITING A BOOK which deals with the industrial applications of a scientific technique there are three possibilities afforded to the author. Firstly, he may decide to cover the subject fully, in which case he is very unlikely to be thoroughly familiar with more than a small part of the whole technology, consequently he has to digest other books and many papers recording original work relating to subjects of which his knowledge is not gained from first hand experience. This method leads to a good book if the author has wide experience, a strong critical faculty, good imagination and infinite patience. Secondly, he may collect together a team of writers who have, collectively, a comprehensive knowledge gained from practical experience. This approach is becoming more and more widely adopted, especially in America, but it often results in a work lacking homogeneity. Thirdly, the author may write fully about those aspects of the subject of which he has great experience, and neglect or deal summarily, with technologies outside his special province. This last course has apparently been adopted by the author of *Industrial High Vacuum.* As a result the work is out of balance and might even be said to have a misleading title. Nevertheless, it is a valuable book.

The author is manifestly an authority on the deposition of metals and salts in vacuum, and at least half the book is devoted to detailed accounts of this work. The information given on this subject is first-class : a great deal of data is given in book form for the first time, and many results of the author's own experimental work are included.

On the other hand, despite the rapid growth of vacuum science in many new fields of technology in recent years, the manufacturers of electron tubes are surely still the largest users of vacuum apparatus. This book, however, confines the details of this vast use of vacuum to a few miscellaneous details here and there in the text, supplemented by a three page appendix called ' Emission of Electrons : Cathodes.'

Again, there is little in the work to awaken the interest of those new users of large-scale vacuum plant, the physicists and engineers undertaking developments in nuclear physics. Apart from the extensive details on vacuum deposition, however, the book includes chapters on 'Molecular Distillation,' 'Vacuum Metallurgy'—the only practical account of this growing subject which has so far appeared in a technical book—and on 'Vacuum Dehydration.' These three sections are well written, but the last two of them suffer from inadequate accounts of complete apparatus. Again, the number of references to original work is inadequate. For example, in the chapter on dehydration, there is no mention of the valuable work done by Flosdorf in America and by Hargreaves in England. The intensive efforts of manufacturers to develop apparatus in these fields is also unrecognised.

The introductory chapters deal with general principles, vacuum pumps and gauges. Pressure measurement practice is considered in outline with simple line drawings of equipment. This seems a pity in a book on industrial practice, since details of the excellent work which has been done on improving the stability and facility of operation of gauges like the Pirani and the ionisation types would have been invaluable. The chapters called 'The Working Chamber' and 'Department and Layout' are well written and contain much sound advice. However, they are concerned exclusively with vacuum deposition processes. Other chapters are on 'Seals and Gaskets'—an interesting and useful account, but two references only seem a poor reward to the host of workers who have devised various pieces of gadgetry in this field—'Valves and Interlocks,' 'Mechanisms in Vacuo' and 'Leads, Heaters and Heater Supplies' – the valuable technique of using radiation shields is overlooked in the last of these sections. Chapter 9 is entitled 'The H.T. Unit and the Discharge Operation': it contains much useful and original data on the value of the ionic bombardment method of cleaning up the vessel and glasses prior to vacuum deposition. The chapter 'Leaks and Leak Finding' is sadly lacking as a balanced account. It deals excellently with the use of a discharge tube as a detector of leaks and includes schedules for leak location procedure using this instrument. But there is nothing about the use of the Pirani gauge in leak detection, or about the hydrogen sensitive ionisation gauge and the halogen leak detector, whilst the details of the mass spectograph on page 92 are surely inadequate. Such instruments are invaluable in leak detection techniques and should find a place in a book with the title of the present one.

Chapters 14 to 19 inclusive are devoted entirely to thin films and their deposition *in vacuo*. This is the best account of this useful technology extant. Any scientist or technician undertaking such processes will profit by reading Mr. Davy's book. Full details are given of the preparation of opaque and semi-transparent films of all the useful metals and on the theory and production of anti-reflection films, including sandwich layers. A minor comment here is that some diagrams with discussions of complete plants would have added to the value of the book.

In general, the data given in the book is sound and placed on a good scientific basis. There are, however, a number of assertions in the text with which the reviewer disagrees. Examples of these are that on page 4 it is stated that the mean free path for air at 0.001 mm. Hg is 5 mms.; it should be 5 cms. Again, it is doubtful whether silicone oils replacing Apiezon oils in a diffusion pump result in a faster pump if the heater wattage is the same. In fig. 7a the side tube labelled 'To Gauge' is in the wrong place; it is important in pumping speed measurements to avoid having the gauge connected in the air inlet line before the main vessel. It is not true, as stated on page 20, that gettering is carried out in radio valve manufacture *after* the roughing pump tap is closed. The getter still acts as a contact adsorber after the valve is sealed from the pumps, but the getter is fired whilst the valve is still on the rotating pump table. In discussing the rotary pump it is stated that the vacuum obtainable in a tight system should be at least 0.001 mm. Hg. Yet manufacturers' single-stage pumps, which are widely used, only give an ultimate pressure of 0.005 mm. even when a McLeod gauge is used to measure the vacuum. On page 90, the author suggests that they (thermistors) 'would repay investigation for high vacuum purposes.' But thermistor gauges have already been constructed and are discussed in some detail in 'Scientific Foundations of Vacuum Technique' by S. Dushman (1949).

The book is well written, the author evincing an excellent command of language, and his enthusiasm for his subject is well conveyed. There are eighty-two well drawn illustrations.

J. YARWOOD.

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. . . of vacuum interest

The Editor will be pleased to consider for publication in this column news items and personal notes of interest to vacuum workers.

Lectures

NORWOOD TECHNICAL COLLEGE, Knights Hill, West Norwood, London, S.E.27, is organising a lecture course on vacuum technology to be held once a week during the session period 1952-1953 and commencing on September 22nd, 1952. The lecturer will be Mr. J. Cooper, B.Sc., a member of the college staff.

The lectures will cover general vacuum physics, the production and measurement of low pressures, techniques of outgassing, leak detection and vacuum plumbing. In addition, applications of vacuum techniques will be discussed, particularly in the fields of physics, electronics, metallurgy, chemistry and biochemistry.

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FURTHER LECTURES are announced as follows :

November 13th 1951 at 5.30 p.m.
The Electrical Properties and Structure of Evaporated Films
Lecturer : *F. Ashworth.*
Held at : 47, Belgrave Square, London, S.W.1.
Arranged by : The Institute of Physics.

December 21st 1951 at 7.30 p.m.
High Vacuum Technique.
Lecturer : *J. Blears.*
Held at : The College of Technology, Manchester.
Arranged by : The Society of Instrument Technology.

January 24th 1952 at 7.30 p.m.
High Vacuum in the Service of the Chemist.
Lecturer : *L. G. Beckett.*
Held at : Chemistry Lecture Theatre, Bristol University.
Arranged by : Bristol Branch of the Royal Institute of Chemistry.

April 18th 1952 at 7 p.m.
Vacuum Pumps and Techniques.
Lecturer : *D. Latham.*
Held at : Reynold's Hall, College of Technology, Manchester.
Arranged by : The Institute of Electronics.

Licensable Patents

THE NATIONAL RESEARCH DEVELOPMENT CORPORATION* is an autonomous corporation established by the Board of Trade in 1949 under the Development of Inventions Act, 1948. It is responsible for the exploitation and where necessary development of inventions which result from the expenditure of public funds. Inventions which are primarily of defence, jet propulsion or nuclear energy nature are outside its normal activities.

Under current arrangements particulars of inventions evolved in government laboratories, such as those of the Ministry of Supply, the Department of Scientific and Industrial Research (D.S.I.R.), the Medical Research Council and other organisations carrying out research with the assistance of public funds, are notified to the Corporation shortly after the filing of relative patent applications and the title thereto is subsequently assigned to the Corporation which acts as principal in arranging manufacture under licence.

The Corporation's portfolio of inventions now comprises some 1,000 patents and patent applications. During the past 12 months particulars of more than 300 patent specifications have been circularised by the Corporation in each case on the average to about 25 appropriate firms potentially interested in its exploitation.

'Vacuum' is glad to assist the Corporation in their efforts to reach an even wider circle of manufacturers likely to be interested in the exploitation of inventions assigned to that body which have a bearing on vacuum technology, and will publish from time to time general descriptions of patents in this category. The corporation has sent us particulars of the following three patents for inclusion in this issue :

Patent No. 639,281. *A Continuously Indicating Gauge for Pressure of the Order of* 10^{-8}*mm.Hg.*

This gauge is essentially a fast operating oil-diffusion pump across which is shunted a flow resistance system. The pressure difference which develops across the latter can be measured either directly by an oil manometer or via a metal bellows arrangement. This invention is effectively a continuously indicating McLeod gauge and is intended for use with large systems. The time response

* Enquiries can be addressed to : Dr. B. J. A. Bard, Industrial Manager, The National Research Development Corporation, 1 Tilney Street, London, W.1, England. (Tel. : Grosvenor 5431).

(about $\frac{1}{4}$ minute) depends upon the speed of the pump ; the variation of the sensitivity for relatively large changes in rates of heating and cooling is about 5%.

Pat. Appln. No. 3086/50. *A Controlled Vacuum Coating Device*

The electric current passing between the vaporising filament and an adjacent electrode maintained at a suitable potential within a conventional coating chamber, is used for control purposes. At an operating pressure of 10^{-6}mm.Hg, the electron emission from a tungsten vaporising filament is quite adequate to operate a relay via which a shutter can be operated to permit coating of subjects only under predetermined conditions or to control the rate of feed of the material to be vaporised.

Pat. Appln. No. 12504/50. *'Shadowless' Coating*

At 10^{-4} to 10^{-6}mm.Hg the mean free path of molecules of coating materials is sufficiently large to cause 'shadowing' on irregular and re-entrant subjects. If the pressure within a conventional coating-chamber is increased by the introduction of an inert or chemically unreactive gas, the mean free path can be so reduced that shadowing is substantially eliminated, but the mean kinetic energy is still great enough to obtain good coherence. Optimum operating pressures depend upon the coating material and its rate of evaporation, the nature of the introduced gas and the geometrical arrangement of the objects within the chamber. With argon, the pressures necessary for coating copper and silver range from 0.5 to 0.05 mm.Hg.

Miscellanea

VACUUM PHYSICS is the title of a supplement to the 'Journal of Scientific Instruments' published by the Institute of Physics this month. It contains 11 papers presented at a symposium arranged by the Midland branch of the Institute of Physics and held on the invitation of Professor M. L. Oliphant at the University of Birmingham from the 27th to the 28th June last year. The lectures cover a wide range of subjects related to vacuum technology and deal with the design and operation of plant and major applications of vacuum processes in the fields of electronics, metallurgy and bio-chemistry.

The following authors have contributed :
D. R. Goddard on Modern Vacuum Pumps.
T. S. Millen on The Design of Industrial Vacuum Systems.
W. Steckelmacher on Vacuum Gauges.
J. Blears and J. H. Leck on the General Principles of Leak Detection.
C. J. Milner on A Cold-Cathode Mass Spectrometer Leak Detector.
J Blears on the Application of the Mass Spectrometer to High Vacuum Problems
D. R. Goddard on Large Scale Vacuum Dehydration.
L. Riddiford on The Vacuum System of the Birmingham Proton Synchrotron.
L. Holland on Recent Advances in Vacuum Coating Plant and Techniques.
E. D. Malcolm on Melting and Sintering of Metals in Vacua.
L. G. Beckett on Freeze Drying.

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THE PRESERVATION OF THE DECLARATION OF INDEPENDENCE and the Constitution of the United States is the subject of a report published by the U.S. Dept. of Commerce (National Bureau of Standards Circular 505) a few months ago. Both documents are on display in a Shrine at the Library of Congress and provisions have been made recently to safeguard the animal parchment on which they are inscribed against deterioration for an indefinite period. To achieve this, oxygen cannot be tolerated in the atmosphere surrounding the documents. The ideal would have been to provide for a perfect vacuum, but as it is difficult to maintain even a partial vacuum for any length of time in the circumstances, it was decided to fill the container housing the documents with an inert gas. Another point that had to be taken into account was the moisture problem. The main constituent of the parchment used for the documents is a protein compound, Collagen. Extensive studies on the behaviour of Collagen were conducted and as a result the inert gas was artificially moistened when filled into the casing, to give an atmosphere with a relative humidity of 25-35% at room temperature inside. Helium was the inert gas chosen as it is commercially obtainable in a very pure state. In addition helium has a relatively high thermal conductivity compared to air. This property of helium has been employed for the detection of air which may have leaked into the container. Four thermal conductivity cells are used connected to form the equal arms of a Wheatstone bridge. Two of the cells are filled with helium and sealed to act as reference cells and two are placed inside the casing to act as measuring cells. The documents are resting on a backing paper which is of pure cellulose and serves as a humidity reservoir to offset any changes in the water vapour content of the helium atmosphere caused by the variation of ambient temperature.

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VACUUM
CLASSIFIED ABSTRACTS

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

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14 — MECHANICAL SCIENCES
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20 — PUMPS
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29 — MISCELLANEOUS APPARATUS

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
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Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) a Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

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10 — VACUUM ENGINEERING (GENERAL) — 10

Progress of Vacuum Technology in the Years 1949 and 1950 86/I

Germany. The article consists essentially of a bibliography covering recent publications related to vacuum engineering. The information presented is classified under seven headings as follows :—

(1) General
(2) Production of Vacuum
(3) Measurement of Vacuum
(4) Technical Data and Accessories
(5) Physics
(6) Chemistry and Biology
(7) Methods of Applications

In his concluding remarks the author points to two developments in particular, the progress of research on vacuum polarisation and the establishment of new techniques for the measurement of radiation at high altitudes. Some instruments used for the latter purpose are no longer exhausted by ' artificial ' means on the ground. Their evacuation is effected ' naturally ' as they ascend to the required height.

Sommaire : Une bibliographie en deux parties, qui cite les publications récentes reliées à la technique du vide, classée en 7 chapitres.

Article by H. Ebert *Glastech. Berichte* **24**, June & July 1951 152-157 & 177-178

The Technical Use of High Vacuum 87/I

United Kingdom. An account is given of the development of vacuum engineering in the last 30 years. Rotary and diffusion pumps are discussed and the steadily increasing demands placed on their capacity due to the progress made in the field of electronics and atomic energy. The author then turns to industrial applications and gives an analysis of the advantages associated with processing *in vacuo*, generally.

Sommaire : Revue généralisee des derniers développements de la technique du vide et ses applications industrielles.

Article by E. N. da C. Andrade *The Times (Science Suppl.)* April 1951 3, 5-6

Power from the Sea's Temperature 88/I

See Abstract No. : 97/I

Vacuum – The Magic Hat of Industry 89/I

United States. This is a review of the uses and benefits vacuum has to offer to industry as a whole. The following processes are discussed in some detail : food processing (in particular milk and coffee), drying of blood plasma, purification of penicillin, molecular distillation, vacuum refrigeration, all kinds of vacuum coating procedures, extrusion methods used in metallurgy, the separation of metals, the etching of metals, vacuum impregnation and, finally, experimental methods employed in the field of ballistics simulating conditions at high altitudes. 12 illustrations and two tables are given.

Sommaire : Compte rendu des procédés à vide récemment introduits dans l'industrie.

Article by Anon. *Compr. Air Mag.* 56, June 1951 142-149

On the Initial Decay of Oxide-Coated Cathodes 90/I

See Abstract No. : 66/IV

Efficiency and Mechanism of Barium Getters at Low Pressures 91/I

United Kingdom. The experiments reported here were carried out in a small tubular vessel evacuated to approximately 10^{-5}mm. Hg. All measurements were taken with an ionisation gauge of the type recently described by Metson and incorporating a suppressor shield for the elimination of X-rays. Barium was employed as the gettering material and its performance studied with respect to the following gases : oxygen, nitrogen, hydrogen, carbon monoxide, carbon dioxide and water vapour. The first series of experiments was designed to determine the gettering rates. It was found that the gettering rate in an oxygen atmosphere was very high, approximately 700 cm^3/sec., and largely independent of pressure for an appreciable period. Nitrogen, on the other hand, was taken up at a lower rate, approximately 300 cm^3/sec. In this case the rate decreased with time and rising pressure. Both these values were found to be typical for the remainder of the gases investigated. The getter performance in oxygen was representative for the performance in carbon monoxide, carbon dioxide and water vapour atmospheres and conditions applying to nitrogen were typical for hydrogen. All measurements were taken with the manifold at a constant pressure and with the ionisation gauge operating all the time. Whenever the ionisation gauge was switched off, a marked influence on the gettering activity could be noticed. The author's observations very largely coincided with those of Haase who employed a Knudsen gauge in his experiments and established that no gas at all was taken up by a barium getter in the pressure region of 10^{-8} to 10^{-6}mm. Hg, if the getter was evaporated and used under normal conditions. Thus, it must be assumed that the high gettering rates, recorded with the ionisation gauge ' on,' are essentially due to the taking up of products of interaction between the gas molecules and the electrons passing from one electrode of the gauge to the other;

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i.e. a form of ' discharge gettering ' is taking place. Investigating these conditions further the author comes to the conclusion that the gettering effect is mainly produced by uncharged particles. In a final series of experiments it is investigated whether the position of the getter relative to the area where the discharge takes place has an influence on the effectiveness of its performance. Oxygen, it was found, is taken up at a constant rate largely independent of the distance of the getter from the discharge area, however the clean-up of nitrogen is deteriorating as the distance of the getter from the discharge area is increasing. The observations in the case of oxygen seem to confirm that the particles mentioned are atoms, but in the case of nitrogen they appear to consist of metastable molecules formed in the discharge area. These metastable molecules will reform preventing the getter from being effective if sufficient time is allowed, i.e. if the getter position is sufficiently far away from the discharge area.

Sommaire : Le mécanisme et les performances de getters au barium dans différentes athmosphères gazeuses sont discutés.

Article by S. Wagener *Brit. J. Appl. Phys.* **2**, May 1951 132-138

Vacuum Valve for the Handling of Very Pure Gases 92/I

See Abstract No. : 154/II

Glass Valves for Vacuum Control 93/I

See Abstract No. : 153/II

Freeze Drying 94/I

United Kingdom. A review article on the merits and uses of freeze drying methods in various fields. Some details of the technique are given. During the last 2½ years of the war more than half a million bottles of blood plasma and serum were dried in the plant operated by the Medical Research Council which met the total requirements of the Royal Navy, the civilian population, the home needs of the Royal Air Force and a substantial part of that of the Army. The preservation of virus, bacteria, moulds, etc. is mentioned. The possibilities of tissue drying is discussed. Finally, the author comments on the potential importance of freeze drying to the food industry. Attention is drawn to a Symposium on Freezing and Drying which has recently been organised by the Institute of Biology embracing the subject of freeze drying in a great number of lectures published on that occasion.

Sommaire : Compte rendu sur les avantages des méthodes de lyophilisation.

Article by E. Hindle *The Times* (*Science Suppl.*) Aug. 1951 9

Vacuum Metallising 95/I

See Abstract No. : 147/III

Metallising Glass 96/I

See Abstract No. : 152/III

13 — ELECTRICAL SCIENCES — 13

Power from the Sea's Temperature 97/I

France. A report is given of an experiment now in progress at Abidjan on the Ivory Coast of West Africa. An electrical power plant is being built which derives its energy from the temperature difference between the surface and the deep waters of the ocean. In tropical countries this difference amounts to 36°F between the surface and a depth of 1,300 ft. The warm water is pumped into an evaporating chamber connected with the housing of the turbine which drives the generator. To facilitate the evaporation of the incoming water without raising its temperature artificially, the whole plant is kept under sub-atmospheric pressure. The vapour leaving the chamber drives the turbine and enters another chamber where it is condensed by contact with cold water brought up from the deep sea. A thermal station is being erected on the same site to ensure continuity of power generation, should at any time the temperature difference between surface and deep sea waters be insufficient to maintain the operation of the main plant. It is expected, however, that the main plant will operate satisfactorily for 10 months in every 12, i.e. it will be just as reliable as hydro-electric power stations which are affected by droughts or other climatic irregularities. The plant under construction at Abidjan consists of two units of 3,500 kW each. It is estimated that an installation of typically commercial size, say 15,000 kW, would require 4,000 kW for pumping the water—9,000 gallons of warm water and 3,100 gallons of cold water per second—and 1,000 kW for the evacuation of the plant. Thus a 15,000 kW turbine would yield a net output of 10,000 kW.

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Sommaire : Des essais pratiques sont faits en vue de l'utilisation de la différence de température entre les eaux profondes et celles de surface de l'ocean pour la production de courant électrique. Le vide est employé pour faciliter l'ébullition à la température de l'eau de mer de surface.

Article by Anon.
The Times (Industr. Suppl.)
May 1951, 24

Electrical Breakdown over Insulators in High Vacuum

98/I

United States. Phenomena described in this article are of interest regarding insulation conditions in high vacuum generally. The author based his investigation on research work carried out by Korfoid. He used Pyrex glass insulators in the form of cylinders or rods held between two copper electrodes. This assembly was placed in a vacuum chamber made of glass and measuring 11 × 9 inches which could be evacuated with 4-inch diffusion pumps employing Litton oil. All measurements were carried out at a pressure below 10^{-5}mm. Hg. No cold traps were used. Details of the experimental procedure are given. The investigation proved that the pressure prevailing in the chamber had no effect on the breakdown voltage as long as its value was below that, at which glow discharges occurred. In an endeavour to trace the origin of the charged particles initiating the breakdown, measurements were taken of pre-breakdown currents. The results of these measurements—5×10^{-11} amps. in some instances and 5×10^{-7} amps. in others—were not conclusive. Geiger-Muller counters were used to detect X-ray radiation and two pin-hole cameras to assess the energy and distribution of that radiation. The actual breakdown current was observed with an oscilloscope. The arc-like discharge extinguished, when the current reached the value of 1 amp. If the resistance in the circuit was increased and the current kept below the maximum value of 1 amp., no stable discharge developed. Breakdown voltages dropped to 2.5 kV in some cases and to less than 100 V in others. A series of breakdowns over the same insulator caused certain ' conditioning ' phenomena, some of which were permanent. See Abstract No. : 99/I.

Sommaire : Le mécanisme de l'effondrement électrique d'isolateurs sous haut vide a été étudie.

Article by
P. H. Gleichauf
J. Appl. Phys.
22, May 1951
535-541

Electrical Breakdown over Insulators in High Vacuum

99/I

United States. The present paper is a continuation of a report published by the same author previously (see Abstract No. : 98/I). Here, the question is examined whether certain properties of various insulating materials such as fused quartz, pyrex glass, polystyrene, teflon, etc., affect the value of the breakdown voltage. It has been found that the dielectric constant and the density of a material have little effect. On the other hand, surface resistivity of the insulator seems to have some effect. It has been observed that the breakdown voltage increases as the surface resistivity grows. Also, roughening the intervening surface of a glass or fused quartz insulator between the electrodes resulted in a 40% rise of the breakdown voltage. Finally, the influence of the length of the insulator was investigated and conditions as they apply, if insulator and vacuum gap are in series. All experiments were carried out in a system evacuated by oil diffusion pumps but no cold traps were used. Detailed results are given in the form of graphs.

Sommaire : Rapport concernant des essais faits avec le but d'étudier le comportement de différents isolants dans le vide.

Article by
P. H. Gleichauf
J. Appl. Phys.
22, June 1951
766-771

Igniting an Arc for Spectrographic Analysis in Vacuum

100/I

United States. Striking an arc in air at atmospheric pressure is usually effected by pushing the electrodes together and subsequently separating them by hand. This method can only be applied to the striking of an arc inside an evacuated vessel, if an expensive seal is provided through the wall of the vacuum chamber. An arrangement is proposed which effects the separation of the two electrodes by gravity action, i.e. automatically. In the case considered an arc had to be struck *in vacuo* between two vertical electrodes, the lower one carrying a small slab of titanium on its tip. The bottom electrode could move in a sleeve and by means of a Wood's metal pin, it could be held in a raised position where it touched the top electrode. Current passing from the upper electrode heated the titanium slab. The heat generated in the slab was conducted down through the electrode to the pin. In a few seconds the latter melted and the lower electrode dropped to the bottom of the sleeve.

Sommaire : Il est donné une méthode pour amorcer un arc sous vide évitant la nécessité de joints compliqués etc.

Article by
A. J. Mitteldorf
J. Opt. Soc. Amer.
41, Sept. 1951
646

The Effect of Oxide Impurities Upon the Initiation of Arcs

101/I

United Kingdom. In the course of experiments on arcing phenomena in hydrogen at low pressure, the tungsten electrodes were placed 3.9 mm. apart and the pressure was reduced to 10^{-3}mm. Hg. Under these conditions an arc would strike at 210 V supply voltage. However, this figure was considerably lower, i.e. 145 V, if small traces of an oxide, such as calcium oxide, were present on the cathode surface. It appears, therefore that oxide mpurities on the cathode surface improve the conductivity in the gap between the electrodes. This agrees with findings by Llewellyn Jones and Davies, who showed that a thin oxide film on the cathode can produce enhanced pre-breakdown electron emission.

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Sommaire : Les effets d'impuretées d'oxydes, présentes sur les faces de cathodes, sur l'amorçage d'arcs à faible pression.

Letter by F. H. Newman *Brit. J Appl. Phys.* **2**, Oct. 1951, 303

Normal Cathode Fall and Striking Voltage as a Means of Determining Residual Moisture in Air in Connection with Vacuum Drying Procedures

See Abstract No. : 116/I

102/I

Checking the Quality of Aluminised Screens

103/I

United States. The introduction of aluminised phosphorus screens in T.V. tubes required a change in the procedure for the testing of phosphorus screens in production. The conventional method provided for a high-frequency spark coil to be moved across the faceplate of the tube carrying the phosphorus screen, but the aluminium film in the new designs acts as a radio-frequency shield and consequently the phosphorus screen does not light up. There are two alternative methods for testing phosphorus screens backed by aluminium films. The first provides for the neck of the tube to be joined to a vacuum system and evacuating the tube to a pressure of approximately 5 micron Hg. A metal probe which is introduced into the tube prior to pumping is connected to a high-frequency oscillator and sets up an electrical field between the probe and the aluminium coating. As a result ionisation takes place in the conical part of the tube. During the period, the charge is positive, the ions are repelled by the probe and move in the direction of the screen : the screen assumes a positive potential with respect to the ground. During the negative half of the cycle, electrons and negative ions are striking the screen with an energy sufficiently high, to cause the electrons to penetrate the aluminium film and excite the phosphorus. Details of the second method which uses d.c. of 10,000 V as a source of ionisation are given as well but this method is not often used in practice.

Sommaire : Deux méthodes de contrôle des qualités des écrans phosphorescents de télévision ont été développées.

Article by H. J. Evans *Electronics* **24**, July 1951 140 & 184-188

Metallising Glass Components for T.V.

See Abstract No. : 143/III

104/I

15 — PHYSICS — 15

Friction of Clean Metals and the Influence of Adsorbed Films

105/I

United Kingdom. A series of experiments was carried out (a) to investigate the frictional behaviour of two metallic surfaces which have been thoroughly cleaned and (b) to ascertain the influence of gases adsorbed by such surfaces on frictional behaviour. The apparatus used in the experiments consisted of a quartz glass tube which could be evacuated by a pumping unit comprising two silicon oil diffusion and one rotary pump. The metal specimens used were of cylindrical shape with a flat area on one side. Both specimens required for one experiment were attached to quartz glass rods. The support of one of the specimens was connected to a spring. The longitudinal deflection of the latter could be photographed and this facilitated the preparation of plottings giving absolute friction values. The end of the glass rod supporting the other specimen carried a bulb filled with iron powder. By moving an electrically energised coil along the outside of the vacuum envelope the second specimen could be caused to rub against the first. The weight exercised during the rubbing motion was determined by the weight of the specimen. The degassing and cleaning of the two specimen surfaces was carried out *in vacuo* at high temperatures, the two specimens being kept apart during the process. The actual friction experiments were carried out at pressures of 10^{-6}mm. Hg. The following metals were studied : nickel, iron, platinum and uranium, and the following gases were used in the second series of experiments : oxygen, hydrogen, water vapour, vapours of caproic and lauric acids, hydrogen sulphide and chlorine. Generally speaking, it was found that thoroughly cleaned metal surfaces if allowed to touch each other, will seize firmly even at room temperature. The adsorption of gases by the cleaned metal surfaces influenced the frictional behaviour to a varying degree. Where the adsorbed gas reacted with the metal and formed a solid and chemically attached film, as for instance in the case of chlorine, a pronounced lubrication effect could be noticed. Discussing the significance of the experimental results as a whole the authors state that a temperature coefficient of friction for clean metals can have little meaning, since seizure occurs at all temperatures. So far as surface contamination is concerned, they state that the frictional behaviour of metals is very largely governed by the nature of the surface film and by the extent to which such a film can reduce the amount of real contact between two metal surfaces.

Sommaire : Le comportement du frottement de surfaces métalliques soigneusement nettoyées et l'influence de l'adsorption gazeuse sur ce comportement ont été étudiés dans un appareil sous pressions subathmosphérique.

Article by F. P. Bowden & J. E. Young *Proc. Roy. Soc* **208A**, 7.9.51 311-325

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Evaporation and Mobility of Naphthalene Molecules

106/I

France. A letter has recently been published describing experiments concerned with the evaporation of naphthalene molecules in vacuum (see Abstract 76/I). The writer of the present communication agrees generally with the results of these experiments, but questions the numerical values for the surface mobility of the naphthalene molecules arrived at by the author of the original letter. A mathematical analysis of the problem is given according to which the distance covered by the mobile molecules should be appreciably smaller.

Sommaire : Discussion critique des expériences d'évaporation de molécules de naphtalene sous vide, décrite dans le Sommaire 76/I.

Letter by N. Cabrera *Nature* **167**, 12.5.51 766-767

Measurement of the Thickness of Thin Films by Multiple-Beam Interferometry

107/I

United Kingdom. The accuracy obtainable in thickness measurements of thin films by the multiple-beam interferometry (Tolansky) method has been investigated. The method provides for the deposition of the film to be measured on a smooth substrate in such a manner that a well-defined edge is produced. A highly reflective opaque layer is then deposited on top of the film in an area which includes the edge. Finally, a wedge is formed between the latter and a heavily silvered optical flat and the system examined with parallel monochromatic light by reflection. The wedge is adjusted so that the fringes of constant thickness cross the edge of the film at right angles. The displacement of the fringes crossing the step facilitates the determination of the thickness of the film. The following factors can influence the accuracy of this method: (a) small irregularities in the surface of the supporting plate and of the optical flat may interfere with the normal fringe displacement and (b) it may be difficult to locate accurately the centre of the fringes. Both these factors depend on the wedge angle between test plate and optical flat, i.e. on the number of fringes per centimetre along the step. Optical windows, 2.2×1.1 cm. in area, and optical flats used for the thickness measurements described below were examined for irregularities and found to be flat within $\lambda/150$ and $\lambda/20$ respectively. Details of the testing procedure are given. The errors arising from the displacement of the fringes due to surface irregularities of this type increase with decreasing wedge angle. For any pair of surfaces of known irrularities an optimum value of fringe spacing exists. In the case considered this value ranges from 10 fringes per cm. for film thicknesses of 100 Å to about 15 fringes per cm. for film thicknesses of 2,000 Å. The total error, therefore, will be of a magnitude of 10 to 15 Å. The actual thickness measurements were carried out on test films of silver and of lithium sulphide. Aluminium, nickel, chromium and tin were used as reflecting surfaces. The measurements covered a range of 120 Å to 1,500 Å and all measurements showed good agreement as long as carried out on freshly prepared films. However, ageing of the film had to be taken into account, in particular, in the case of chromium which seemed to accelerate the tarnishing action of the atmosphere on the silver layer below the chromium.

Sommaire : Examen de la précision des mesures d'épaisseur de films minces par la méthode Tolansky.

Article by O. S. Heavens *Proc. Phys. Soc.* **64B**, May 1951 419-425

Determination of the Thickness of a Thin Film Obtained by Condensation of Artificial Radio-Active Elements

108/I

France. A method of measuring the thickness of films of radio-active antimony is described. The films were obtained by condensation from molecular beams on a plate, precautions being taken to ensure a uniform deposit. The activity of the film is measured and compared with that of standards deposited on identical surfaces. These standards were prepared by evaporating a drop of a solution of $SbCl_3$ obtained by the action of *aqua regia* on a known mass of radioactive antimony. The plate was covered with a very thin film of paraffin to ensure that the liquid spread uniformly over the plate.

(*Science Abstracts*)

Sommaire : Description d'une méthode facilitant la mesure d'épaisseur de films minces en antimoine radio-actif.

Note by M. Devienne *C. R. Acad. Sci. Paris* **232**, 12.3.51 1088-1089

Transparent Luminescent Films

109/I

See Abstract No.: 137/III

The Application of Coatings to Optical Elements in Microscopy

110/I

See Abstract No.: 140/III

Reflection and Transmission Infra-Red Cells for Use at Liquid Air Temperatures

111/I

United Kingdom. The observation of infra-red spectra has recently been extended to samples cooled down to very low temperatures. Examination of specimens kept at low temperature gives the opportunity of measuring gases and liquids in the solid state. This is greatly welcomed as it has been shown in the past that a change of physical state can produce variations, e.g. a narrowing of the bands coupled with an apparent increase in resolution. Low-temperature studies of this kind prove somewhat difficult due to the fact that glass does not transmit infra-red radiation and suitable transparent materials must be incorporated in the experimental apparatus to allow for the passage of the radiation. A newly designed reflection and transmission cell are

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described which have given good service in such investigations. The reflection cell consists essentially of a long copper rod fitted at one end with a detachable copper head which carries a stainless steel mirror. At the upper end is a glass extension which is 3 inches high and 2¾ inches wide. Four glass joints are provided for insertion of thermocouple leads, etc. The top is covered with a glass plate which is cut out in the centre and carries a rock salt plate of 5 square cm. The reflecting surface is positioned ½-inch below the top plate. Placing the lower end of this cell into a Dewar flask will produce the necessary low temperature in the cell which can be varied by adjusting the length of the copper tubing inserted in the Dewar vessel. The transmission cell essentially consists of two concentric glass tubes forming a double-walled vessel. The specimen support is attached to the inner wall in such a manner that the specimen is in line with two rock salt plates sealed into the outer wall facilitating the passage of the radiation through the container. Three tubular arms are sealed to the upper portion of the cell for the insertion of the thermo-couples and for evacuation. The successful operation of the cell very largely depends on a good vacuum being maintained in the cell in order to prevent appreciable heat gains. The refrigerant (liquid air) is kept in the inner tube. In conditions of a good vacuum the sample can be held at a temperature of —165°C for a period of some hours.

Sommaire : L'on décrit deux types de cuves construites pour être utilisées dans des recherches spectroscopiques dans l'infrarouge à basse temperature.

Article by L. F. H. Bovey *J. Opt. Soc. Amer.* **41**, June 1951 381-384

Matter Moved by Light — 112/I

Austria. Some 20 years ago a Viennese physicist, Professor E. Ehrenhaft, discovered a phenomenon which he called photophoresis. He claimed that light falling on very tiny particles of a liquid or metal will cause some of these particles to move in the direction of the light and others in the direction opposite to that of the light. This phenomenon has been observed at all pressures above and below atmospheric. Dr. Ehrenhaft has recently reported on another experiment which is discussed at length in this article. A spherical glass bottle containing a small amount of graphite powder is evacuated to a pressure of approximately 10 mm. Hg. Shaking the bottle and placing it into a shaft of light in such a manner that the focus of the rays is approximately in the centre of the bottle, has the result that some of the dust particles will rotate around the light beam in a plane strictly perpendicular to the beam axis for a considerable length of time. It has also been observed that the particles not only revolve around the beam but also in spirals around the path of their own motion. When the light is switched off the particles fall to the bottom of the bottle. A theory has been advanced explaining these phenomena, but this is still disputed.

Sommaire : Le mouvement de petits grains de poudre de graphite attribué aux effets d'une source lumineuse violente est traité et une théorie explicative avancé.

Article by R. G. Rabel *Discovery* **12**, May 1951 151-153

Heat Transfer Through Gases at Low Pressures — 113/I

United States. The heat transfer between a hot and a cold plate situated at various distances from each other in a vessel evacuated to various low pressures has been studied. A tank was used 3 feet long and 2 feet diameter with the hot plate arranged in the centre and one cold plate at each end. Evacuation was carried out with an oil diffusion pump, the minimum pressure attainable being 0.02 micron Hg. The outcome of test runs in air at constant spacing (1.5 in.) of the plates is given in tabulated form for pressures ranging from 10^{-5} to 80 mm. Hg. In the case of 12 mm. Hg the average temperature of the hot plate was 157.8°F, the temperature of the cold plates was 85.4°F. The total heat transferred was calculated to be 100.4 Btu per hour. A complete analysis of the various forms of heat transfer is presented. The experiments have been carried out with helium and air as transfer media.

Sommaire : Etude théorique de transport de chaleur de gaz à faibles pressions.

Article by R. E. Peck, W. S. Fagan & P. P. Werlein *Trans. A.S.M.E.* **73**, April 1951 281-287

Sublimation of Ice in Vacuum — 114/I

See Abstract No. : 60/IV

16 — CHEMISTRY — 16

Further Research Indicated for Dehydrated Foods — 115/I

United States. This is a short editorial on the commercial prospects of dehydrated food. At one time during the last war some 240 million lbs. of dehydrated fruits and vegetables were produced per annum. 375 plants were engaged in the production then. At the end of 1949 this figure had dropped to 60 and has since dropped still further to 35. The hope that dried food would be acceptable to the housewife after the war has not been fulfilled. Renewed efforts are being called for to improve the quality of dehydrated food. A research project is mentioned which is now in progress, of dehydrating food with air at room temperature using a desiccant which increases the moisture carrying capacity of the drying air, but it is suggested to enlarge on this by undertaking research into drying in inert gas atmosphere or vacuum drying at temperatures above freezing.

Sommaire : Perspectives de la consommation d'aliments déshydratés aux Etats Unis.

Note by Anon. *Food Industr.* **23**, Feb. 1951 125-126

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Normal Cathode Fall and Striking Voltage as a Means of Determining Residual Moisture in Air in Connection with Vacuum Drying Procedures

116/I

Germany. The voltage required to strike and maintain a glow discharge in air at pressures ranging from 10 to 90 mm. Hg drops rapidly by about 60 volts, if water vapour is present at a partial pressure of 2% or less. It is very difficult to determine satisfactorily a moisture content of the order of 1% with existing methods and it appears that the observation recorded above would yield a method which could be usefully employed in vacuum drying procedures carried out at pressures from 10—100 mm. Hg.

Sommaire : Il est recommandé d'utiliser le changement de caractéristiques électriques d'une décharge luminescente normale dans la région de 10—90 mm. Hg pour la détermination de très faibles teneurs en humidité dans des gaz et de l'air.

Article by A. Hinzpeter & W. Meyer *Z. Angew. Phys.* **3**, June 1951 216-218

Drying of Pharmaceutical Powders and Crystals

117/I

See Abstract No. : 153/III

Some Physical Factors that Influence the Survival of Brucella Abortus During Freeze Drying

118/I

See Abstract No. : 62/IV

The Method of Shadow-Casting in Photomicrography

119/I

India. The examination of unstained bacteria or protozoa with the usual optical microscope requires the application of a very narrow condenser aperture in order to decrease the obliquity of the illuminating beam and, thus, to increase the contrast of the image against the background. There are cases, where the iris diaphragm fringes appearing around the contours interfere appreciably with the image quality. Staining techniques are often applied to overcome this difficulty but staining is liable to obscure the details of the surface structure of the organisms under test. Shadow-casting techniques are more suitable. The author studied all factors which require attention in producing a deposit satisfactory for the purpose and recommends 50—100 Å to be the thickness of the deposit and $\tan^{-1} 0.25$ to $\tan^{-1} 0.5$ to be the angle, under which shadowing is to take place. The shadowing material should be silver.

Sommaire : Description de techniques de préparation de spécimen faisant appel a l'ombrage par évaporation sous vide pour des bactéries et protozoées déstinées au microscope optique.

Article by D. L. Bhattacharya *Indian J. Phys.* March 1951 145-151

The Densities and Limiting Densities of Vapours

120/I

United Kingdom. The accurate determination of the limiting densities of vapours is difficult to obtain due to large departures from the ideal state and the uncertainty caused by extrapolation to zero pressure. Reference is made to methods employed in the past, all lacking in satisfactory accuracy except for isolated cases. In order to shorten the extrapolation to zero and gain more information on association phenomena in the vapour phase, it is desirable to extend the limiting density method to vapours at pressures as low as possible. The authors describe an apparatus and its performance which consists of a multi-point manometer, a micro-balance, a system for the admission of the sample and means of changing the density of the vapour after admission. The equipment appeared to give reliable results down to pressures of the order of 20 mm. Hg. Evacuation was carried out by a rotary pump. The whole assembly was placed in a thermostatic tank and the temperature of the water was kept constant within a few thousandth of a degree. Details of the construction of the equipment are given. The design of the micro-balance used represented a special development of the adsorption-compensation buoyancy type of instrument. The manometer gave readings which were accurate within 0.002 mm. Hg and the micro-balance had a sensitivity of 0.001 mm. Hg. Four specimens of highly purified benzene from different sources were tested giving largely identical values for the limiting density of the vapour, thus indicating that all four specimens were of a high degree of purity. The molecular weights determined by this method closely agreed with the respective calculated values.

Sommaire : Description de la construction et des caractéristiques d'une micro-balance développée pour la détermination des densités limitantes de vapeurs aux basses pressions.

Article by F. L. Casado D. S. Massie & R. Whytlaw-Gray *Proc. Roy. Soc.* **207A**, 23.7.51 483-495

A Magnetically Compensated Vapour Density Balance

121/I

United States. In the introduction to this article a survey is made of existing methods for the precise and continuous determination of gas and vapour densities falling in the range of 0 to 4 g./l. The authors, when investigating the vapour phase association of carboxylic acids, found that none of the methods or instruments known to them would give sufficiently accurate results. They developed, therefore, a new instrument for the measurement of the densities of mildly corrosive condensable vapours which could be used in a temperature range up to 220°C and at pressures up to 1½ atmosphere. The instrument described is of the magnetically compensated buoyancy type and represents an improvement of the design used by Stock and Ritter. In designing the new instrument special attention was given (a) to facilitating the use of the instrument with chemically active vapours, (b) to widening the temperature range covered by the instrument, (c) to reducing

I — General Science and Engineering — I

Contd.

Abstract No. and References

the super-sensitivity of previous designs and (d) to eliminating the need of frequent re-calibration. Details of the new design are given. The instrument mainly consists of a suitably supported balancing beam incorporating a permanent magnet in its centre and fitted with a sealed buoyancy bulb at one end of the beam and a perforated counter-poise bulb at the other. The balancing assembly is enclosed in a glass envelope and placed above an electro-magnet. The whole assembly could be isolated from the vacuum pump by a valve. Between this valve and the actual balance a condensation bulb and a number of removable ampoules are joined to the system. In operation, the whole apparatus was evacuated and sealed off. Then, the vapour to be examined was introduced through the condensation bulb and all measurements were taken. If a change in pressure was required, part of the vapour was condensed in a removable ampoule and the latter subsequently sealed off. In this manner readings could be taken at a number of different pressures. The sensitivity of the instrument varied from 0.0005 g./l. per mm. scale deflection at 80°C to 0.0009 at 200°C. This allowed for the detection of a change of density as small as 0.0001 g./l.

Sommaire : Présentation d'un instrument à compensation magnétique basé sur le principe archimèdique permettant la détermination de densités de vapeurs condensables de nature faiblement corrosive à des températures jusqu'à 200°C et à des pressions jusqu'à 1.5 athmosphère.

Article by E. W. Johnson & L. K. Nash *Rev. Sci. Instrum.* **22**, April 1951 240-244

Equilibrium-Phase Vaporisation Of Petroleum Crude Oils or Fractions 122

See Abstract No. : 166/III

How to Get More Gasoline from Heavy Fractions 123/I

United States. Reference is made to a method incorporating a high vacuum distillation process which is being developed at the present time with the aim of preventing too many fractions going into fuel oil. The new high vacuum distillation process will recover a greater volume of high boiling hydrocarbons. It is combined with a mild thermal cracking or visbreaking process applied to the highly viscous residue in order to obtain more catalytic cracking feedstocks for greater yields of gasoline.

Sommaire : Utilisation de la distillation sous vide pour la séparation de fractions lourdes d'huiles combustibles.

Abstract by Anon. *McGraw-Hill Digest* **6**, Sept. 1951 37 see also *Oil & Gas J.* **50**, 14.6.51 79

First Double-Flash Tar Distillation Units 124/I

See Abstract No. : 165/III

19 — SCIENCES (other than the above) — 19

The Preservation and Clinical Use of Freeze-Dried Bone 125/1

United States. Bone graft surgery is steadily growing in its importance. Much of its success depends very largely on proper methods for the preservation of bones. The bone banks established so far utilise ordinary freezing methods for the purpose. But there are certain economical and clinical disadvantages connected with this form of preservation. The authors report on a large scale exploratory experiment which was conducted with the object of establishing the merits of freeze drying techniques in this field. Going by results in other medical applications, drying from the frozen state minimises protein denaturation and lessens harmful concentration of salts as the solid state is maintained throughout desiccation. Finally, bone preserved in this manner can be stored at room temperature. In this experiment bones were treated by freeze drying methods at –40°C and subsequently stored for periods up to 122 days at room temperature. For the purpose of comparison, a number of bones were treated by the conventional freezing method and stored in sealed jars at –15°C for an average period of 29 days. Freeze-dried bone and frozen bone prepared in this manner were placed in dogs and their acceptance by and incorporation in the bone system of the animal was checked after a period. The results of these tests seemed to justify clinical use of freeze-dried bone. The freeze-dried bone was somewhat superior to frozen bone with regard to the rate of healing in the early stages. In addition, storage at room temperatures, prolonged storage period and ease of transportation appear to make freeze-dried homogenous bone suitable for use in bone banks.

Sommaire : Détails d'un essai de grande envergure pour déterminer, si les techniques de lyophilisation conviennent à la conservation d'os.

Article by F. P. Kreuz G. W. Hyatt T. C. Turner & A. L. Bassett *J. Bone & Joint Surgery* **33A**, Oct. 1951 863-872

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II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

A High-Speed Hot Baffle for Oil Diffusion Pump Systems — 126/II

United States. Details are given of a device which is intended to stop the migration of oil vapour from the pump to the vacuum system more effectively than baffles of the conventional design. It consists of an electrical heater element inserted into the manifold near the mouth of the pump and kept at a temperature of 650°C. Oil vapour striking the hot surface is immediately decomposed into permanent gases, such as hydrogen, and non-volatile carbon. The new device remains active over the whole of its surface whereas the baffles ordinarily used tend to become saturated in operation. The pressure of the decomposition products of the oil vapour striking the hot surface totals less than 10^{-9}mm. Hg.

Sommaire : L'on donne des détails d'une chicane chauffée jusqu 'à 650°C et qui décompose les vapeurs d'huile diffusant de la pompe à huile dans le système à vide.

Note by R. L. Longini *Rev. Sci. Instrum.* **22,** May 1951 345-346

Laboratory Apparatus for the Distillation of Oils for Diffusion Pumps — 127/II

France. Pump Oils of a certain composition have been distilled under vacuum in a laboratory-size column. Pumps running on oil treated in the manner described produced a vacuum of the order of 1×10^{-6}mm. Hg. Prior to the introduction of this process the lowest pressure obtainable was 7×10^{-6}mm. Hg. Details of the method and the equipment required are given.

Sommaire : Description d'un procédé de distillation de laboratoire recommandé pour la préparation de fluides de pompage en partant d'huiles convenables.

Article by M. Azam & M. Ortel *Le Vide* 7, July-Sept. 1951 1063

Silicone Oil Vapour and Secondary Electron Emission — 128/II

United Kingdom. Secondary electron emission in a system evacuated by silicone oil diffusion pumps has been studied. Determining the ' sticking ' potential of a fluorescent powder the initial values obtained were of the order expected but under electron bombardment the secondary emission began to fall off within 30 minutes and the sticking potential dropped from 30 to 1.5 kV. In another series of experiments various surfaces in an ' orbital beam ' electrode system have been investigated. One of the materials examined was a mixture of magnesium and barium oxide. The source of primaries was a tungsten filament. Heating the secondary cathode to 1,000°C and allowing it to cool the secondary emission coefficient dropped from an initial value of 5 to 1.5. A dark deposit could be noticed on the secondary cathode and staining of a similar kind on the anode and glass bulb of the ionisation gauge. The nature of the deposit was not determined. No such effects could be found on thermionic emitters. As a result of these experiments it is believed that silicone settling on the bombarded electrode may be decomposed, but thermionic emitters are too hot to permit the formation of a silicone film.

Sommaire : Les effets d'huile silicone utilisée comme fluide dans des pompes à diffusion sur l'émission secondaire d'électrons dans les systèmes évacués sont étudies.

Letter by A. Lempicki & A. B. McFarlane *Nature* 167, 19.5.1951 813-814

Vacuum Refrigeration — 129/II

See Abstract No. : 169/III

Machined Carbon Products — 130/II

See Abstract No. : 149/II

21 — GAUGES — 21

Vacuum Gauge Using Alpha-Rays — 131/II

France. This is a short note describing the design and performance of a gauge similar in construction to the alphatron gauge. Polonium is used as the source of alpha-particles. The gauge can be employed for pressure readings in the range of 10^{-1} to 10^{2}mm. Hg. It is specially recommended for the measurement of the pressure of corrosive gases, for instance halogens and is, therefore, particularly suitable for use in connection with the production of Geiger counters. Finally, it could serve as an indicator of the partial pressure exercised by a component gas in a gas mixture, e.g. neon in a neon-argon mixture.

Sommaire : Etude de la construction et des performances d'une jauge à ionisation à particle alpha.

Note by G. Gimenez & J. Labeyrie *J. Phys. Radium* **12,** Oct. 1951 64A— 65A

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Abstract No. and References

Pressure Monitoring Device

132/II

United States. The pressure measurement and control in vacuum systems containing highly corrosive gases is a difficult problem. The materials from which conventional gauges are made are badly attacked by reactive gases and ordinarily such gauges become unusable after a relatively short period of time. The use of corrosive resistant material in the construction of a pressure gauge is not always practicable. The design described here exposes only one element of the gauge to the corrosive gas, the pressure of which is to be measured. A chamber which contains a bellows is filled with a non-reactive gas such as nitrogen, whereas the inside of the bellows is connected to the system containing the reactive gas. The indication of the pressure prevailing in the vacuum system is effected by balancing the pressure exercised on the inside of the bellows against the pressure of the non-reactive gas in the chamber surrounding the bellows. The apparatus can be designed to serve as a monitor controlling the pressure in the system automatically.

R. Livingston & U.S. Atomic En. Comm. *U.S. Pat.* 2,567,519

Sommaire : Description d'une jauge à vide utilisable aussi comme régulateur développée en rapport avec des systèmes à vide contenant des gaz fortement réactifs.

A Null-Reading Absolute Manometer

133/II

United States. The most widely used instrument for pressure readings in the mm. Hg range is the liquid manometer containing either mercury or low vapour pressure oil but this type of instrument has two serious disadvantages :—(a) It can not be degassed at high temperatures, and (b) it can not be used for the measurement of the pressure of condensable vapours. As an alternative diaphragm manometers are frequently used. Here again baking out at high temperature is difficult and may lead to the destruction of the diaphragm The authors describe a null-reading manometer of the diaphragm type which has none of the disadvantages mentioned. An application of this instrument in connection with the admission of a controlled amount of very pure gases to a vacuum system is described. The metallic diaphragm is brazed to a Kovar cup which is sealed to the ' clean vacuum system ' to be filled with the gas. The ' manometer ' system is joined to the open end of the Kovar cup and consists of a liquid manometer for direct reading of the pressure, a mechanical pump and a capacity probe, inserted after the ' clean vacuum system ' has been outgassed. The probe is in a position, a few mils away from the diaphragm, and its capacity is compared with that of a standard capacitor, null-readings being obtained when the two capacities are equal. When both sides of the unit, i.e. the clean vacuum system and the manometer system are evacuated, the diaphragm assumes an equilibrium position which is indicated by its capacity to the probe. Filling the ' clean ' system with the gas will require filling the manometer system with air of a comparable amount until the diaphragm re-assumes its null position. The liquid manometer, will then indicate the actual pressure of the pure gas in the ' clean vacuum system.' Details of the construction of the instrument are given.

Note by D. Alpert, C. G. Matland & A. O. McCoubrey *Rev. Sci. Instrum.* **22**, June 1951 370-371

Sommaire : Détails de construction d'un manomètre à membrane, qui supporte le traitement à chaud.

A Null Method Mercury Cistern Manometer

134/II

United Kingdom. Cistern barometers of ordinary design can only be operated over a limited range of reduced pressures. The null method manometer described here covers all pressures within ± 1 atm. This particular construction was originally developed for the calibration of aircraft instruments, but it could be employed as a transportable standard for the precision measurement of pressures within that range, generally. The accuracy of the instrument is 0.01 inch. The mercury cistern can be moved up and down a column and can be clamped to the column at any desired height. The reading limb—the only part made of glass—is connected on one side to the cistern and on the other to the over-flow reservoir. Neoprene tubing is used for the connections as it is not affected by mercury. Two verniers slide along the right and left hand edge of a bar mounted vertically and adjacent to the above mentioned column and engraved with linear scales of inches and tenths of inches. The left hand vernier is used for pressure measurements in excess of one atmosphere and the right hand vernier for subatmospheric pressures. The two scales are displaced from one another by $3\frac{1}{2}$ inches to prevent errors in reading.

Article by K. R. Honick *J. Sci. Instrum.* **28**, May 1951 140-142

Sommaire : Détails d'un manomètre à cuve de mercure, à méthode de zéro, primitivement construit pour l'étalonnage d'instruments de bord, mais qui peut être utilisé comme standard mobile pour la mesure précise dans la région ± 1 athmosphère.

Glass-to-Metal Seal for Vacuum Systems

135/II

See Abstract No. : 68/IV

Calibration of Ionisation Gauges for Various Gases at Low Pressures

136/II

United Kingdom. In the course of an investigation on getter performance (see Abstract 91/I) it was necessary to obtain calibration data for ionisation gauges operating in various gaseous atmospheres such as oxygen, carbon dioxide, nitrogen, and hydrogen. Due to the danger of chemical attack by these gases calibration had to be carried out at very low pressures, i.e. at pressures where the McLeod gauge is no longer sufficiently sensitive. A Knudsen gauge was used as a reference instrument. An ionisation gauge of conventional design and two ionisation gauges of the suppressor type described by Metson elsewhere have been calibrated. One of the latter two had a tungsten cathode and the other an oxide cathode. The measurements were taken in the pressure

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range of 5×10^{-6} to 2×10^{-4}mm. Hg. Results are given in tabular form. The calibration factors C were determined from the readings taken at 5×10^{-5}mm. Hg and expressed in $\mu A/mA \times \mu$. With the exception of water vapour the values of C measured for the three gauges in the same gaseous atmosphere agreed fairly well.

Sommaire : Détails d'une méthode d'étalonnage de jauges à ionisation travaillants en athmosphère de gaz réactifs.

Note by S. Wagener & C. B. Johnson *J. Sci. Instrum.* **28**, Sept. 1951 278

The Absorption of Ultra-Violet Light as a Means of Detecting Leaks in High Vacuum Apparatus and Measuring Subatmospheric Pressures of Gases

137/II

France. The intensity of a beam of ultra-violet light passing through a layer of gas is substantially reduced due to absorption. If the intensity of the light beam is known before it enters the gas layer and is measured by means of a photocell after emerging from the gas layer, conclusions can be drawn as to the composition, density and pressure of the gas penetrated by the beam. The authors, recommend the use of ultra-violet light of 1,850—1,200 Å, i.e. the ' Schuman range ' of ultra-violet radiation. In this range a number of gases show large absorption bands which they do not show in spectra of longer wavelengths. Practical examples are given of methods based on this effect for the measurement of pressures in the mm. Hg range and for the detection of leaks.

Sommaire : Méthode de détermination de pressions et la recherche de fuites utilisant l'absorption de lumière ultra-violette par les gaz.

Article by J. Romand V. Schwetzoff & B. Vodar *Le Vide* **6**, July-Sept. 1951 1046

22 — MEASURING PLANT (VACUUM) — 22

Head for Mass Spectrometer, Permitting the Use of Elevated Gas Pressures in the Ionisation Chamber

138/II

France. The attachment, of bronze, was inserted between the analyser of the mass spectrometer and the gas source which was to be analysed. The gas pressure in the ionisation chamber was 0.01 mm. Hg; in the spectrometer, as measured with a Knudsen manometer and a Philips gauge it was 3×10^{-5}mm. Hg.

(*Chemical Abstracts*)

Sommaire : Description d'une tête de spectromètre de masse permettant l'utilisation de pressions élevées de gaz dans la chambre de ionisation.

Note by G. Philbert & R. Valentin *J. Phys. Radium* **12**, 143-144

X-Ray Shadow Microscope

139/II

United Kingdom. An X-ray shadow microscope for the projection of shadowgraphs from a point source is described. The electron beam is focused by means of an electron lens on a Lenard window which forms part of the chamber wall. The object is situated outside the chamber and is placed as close as possible to the window in order to obtain a large shadow on the fluorescent screen or photographic plate. Experiments conducted with a spot size of 1μ using a single magnetic focusing lens with aperture (to reduce abberation) gave a resolving power of the order of 1 μ.

Sommaire : Description d'un microscope d'ombre à rayons X permettant de placer l'objet à examiner à l'extérieur de l'enceinte évacuée.

Letter by V. E. Cosslett & W. C. Nixon *Nature* **168**, 7.7.1951 24-25

An Improved Electron Diffraction Camera

140/II

United Kingdom. An electron diffraction camera is described which has been designed specially for the purpose of studying the diffraction of fast electrons by gaseous molecules. The object of such investigations is the determination of inter-atomic distances in vapour molecules. Full details are given of the construction of the camera. To obtain useful diffraction patterns it is essential that the electron beam is as fine, intense and homogenous as possible. Further the gas or vapour sample to be examined should be small in order to avoid ' volume effects ' resulting from large-scale spreading of the vapour. The instrument is fitted with an electron gun of conventional design and an optical system comprising a condensing lens and a focusing lens. The vapour is admitted to the diffraction chamber by means of a small vapour jet crossing the path of the electron beam at a right angle. On the opposite side of the chamber is a liquid air trap which condenses the gas after crossing the electron beam. A baffle unit consisting of three baffles is placed between gun and diffraction chamber with the object of preventing the diffusion of the injected vapour into the neighbourhood of the electron gun. The operational pressure is of the order of 10^{-4}mm. Hg. A high-speed pumping system is an indispensable requirement in this type of work. Therefore, five oil diffusion pumps have been provided, two of which are of the silicone oil type connected directly to the diffraction chamber, the third is connected to the baffle unit and the remaining two serve as backing pumps to the former three. A mechanical pump provides the forevacuum for the two backing diffusion pumps.

Sommaire : L'on donne les détails d'un diffracteur d'électrons construit pour étudier la diffraction d'électrons rapides par les molécules de gaz ou vapeurs.

Article by P. W. Allen H. Mackle & L. E. Sutton *J. Sci. Instrum.* **28**, May 1951 144-151

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Methods and Applications of Electron Diffraction in Industrial Research

141/II

Germany. Theory and mechanism of the diffraction of fast electrons is discussed. Instances are quoted of research work in the field of applied physics, the successful conclusion of which is largely due to the research facilities offered by electron diffraction methods. Details are given of plant requirements, specimen preparation techniques, standard substances suitable for calibration purposes and photographic material. The greatest importance of electron diffraction methods lies in the field of structural investigations of thin layers, ground, polished and corroded surfaces, wear and tear resistance of surfaces and the effects of friction and lubrication. Apart from physics and chemistry, electrical and electronic engineering, organic chemistry and the medical profession have benefited most from the introduction of this new research tool. 552 references.

Sommaire : Revue de la théorie du mécanisme et des applications des phénomènes de diffraction électronique.

Article by A. Stahl *Z. Angew. Phys.* **3**, Sept.-Oct. 1951 349-359 & 382-395

Studies of Thin Films by Electron Diffraction

142/II

United States. In the course of experimental work on the preparation of samples for electron diffraction powder patterns, a new method for the preparation of metal specimens has been developed. The method of preparation described here yielded diffraction patterns consisting of a greater number of rings than usual and giving at the same time a better contrast than hitherto obtained at a critical thickness. A water-soluble plastic film (Elvanol) has been used as a substrate for the specimen. A 15% acqueous solution of this material was spread evenly on a flat piece of glass. After drying, the film was peeled off and stuck to a support using moisture as a binder. The film and the support were placed in a vacuum chamber and the metal (in this case aluminium) was deposited on the Elvanol film by evaporation at a pressure of less than 10^{-4}mm. Hg. After completion of the coating process, the coating and substrate were peeled off the glass plate and placed on a water surface in order to dissolve the Elvanol film. The substrateless specimen was picked up with a screen and placed in the electron diffraction camera. The diffraction patterns obtained from specimens prepared in this manner are shown in three photographs.

Sommaire : Description d'une méthode de préparation de spécimens métalliques pour l'examen par la diffraction électronique.

Letter by N. R. Mukherjee & O. Row *J. Appl. Phys.* **22**, May 1952 681

On the Structure of Graphite Oxide Soot

143/II

See Abstract No. : 147/II

Electron Investigation of Glass

144/II

Germany. The examination of glass films (thinner than 0.1μ) in transmittant electron light, and the replica technique are described in their fundamental results; the shadow-casting method is additionally important in glass electron microscopy. For the study of devitrification phenomena and of the nature of gold-ruby glasses, the results of Bayersdorfer's previous studies are discussed. The electron-diffraction pattern of SiO_2 glass above 1,000° to cristobalite is given as an example for the high accuracy of this method. Also the etching of glass by HF vapours and the fine structure of conchoidal fractures are analysed with the chacteristic rhythms appearing in gold-ruby glass, with gold crystals of only 2μ diameter, acting as centres of mechanical tensions in the glass structure. For the study of the finest details the shadow-casting method is modified by applying a deposit of platinum oxide on the films; the particularly fine-grained nature of this deposit is even beyond the resolution limit of the usual electron microscopes. With such a platinum oxide replica technique the author studied the plastic deformation in the trace of scratches on the glass surface. The structure of reflection-reducing coatings on glass, e.g. of LiF and CaF_2 is also illustrated.

(*Chemical Abstracts*)

Sommaire : Discussion concernant les méthodes et resultats de recherches sur les films minces de verre au microscope électronique et au diffractographe. L'on donne des détails de méthodes de préparation d'éprouvettes.

Article by H. Koenig *Glastech. Berichte* **24**, 1951 167-171

Applications of Polystyrene Particles in Conjunction with Shadow-Casting to the Study of Polished Metallic Surfaces

145/II

United States. The article reports on the preparation of replicas of polished metallic surfaces. The material from which the replica is to be made should be thin and structureless. For the investigation of polished surfaces it is convenient to use negative collodion replicas. All scratches and indentations appear, therefore, as peaks on the replica. As the depth of the scratches is comparatively small, shadow-casting should be employed. The advantages of shadow-casting can be summarised as follows : (1) the determination of the height of elevation is simple, (2) a three-dimensional effect is created, (3) extremely small objects beyond the resolution of the instrument are detected and (4) the thickness of the replica is comparatively unimportant. Chromium, gold and uranium are the most popular materials used in shadow-casting. In spite of the improved conditions for the assessment of height of elevation on shadow-cast replicas, errors can still occur due to the fact that the surface replica is not always in a truly horizontal position on its support. In addition, the local angle of shadow-casting frequently varies from point to point. It is recommended, therefore, to use spherical polystyrene particles, which are known to be exceptionally uniform in size, approximately 2,593 $\pm$ 40 Å, as calibration standards. The material investigated in this particular experiment was large grain 70-30 brass in its annealed condition.

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Various methods of polishing were applied and the results examined in the microscope. Unidirectional polishing under heavy pressure produced scratches of appreciable depth; in fact, it has been noticed that heavy pressure can cause the material near the scratch to flow. Multi-directional polishing, will tend to produce shallower scratches and a sub-division of the whole surface into small discontinuous segments. Difficulties experienced in interpreting the electron micrographs obtained in this investigation are discussed.

Sommaire : Examen des effets de différentes méthodes de polissage de surfaces métalliques au microscope électronique. Il est donné des détails des modes de préparation de spécimens.

Article by I-Ming Feng *J. Appl. Phys.* **22**, June 1951 820-824

Portrait Shadow-Casting 146/II

United States. Conventional shadow-casting procedures produce micrographs with an extreme degree of contrast due to the absence of a metallised face on the shadow side. As a result there is often a real danger of complete obliteration of specimen details on that side. It is, therefore, suggested to metallise the shadowed part of the specimen as well. The amount to be deposited in that area varies and depends on the characteristics of the electron microscope used. As a general recommendation, however, it is suggested to use about $\frac{1}{7}$ of the material required to produce the main shadow for this purpose. The new procedure has the added advantage of giving the specimen greater stability, and providing for better thermal and electrical charge dissipation.

Sommaire : Il est suggéré de déposer une certaine quantité de métal sur le côté ombré d'un specimen ombré à fin d'augmenter la qualité de la micrographie.

Letter by D. E. Philpott *J. Appl. Phys.* **22**, July 1951 982-983

26 — MATERIALS — 26

On the Structure of Graphite Oxide Soot 147/II

Germany. Electron microscopical examination of graphite oxide soot consisting of very thin carbon lamellae coated with evaporated platinum or gold indicated the existence of single crystals of 2,000 Å cross section. Electron diffraction photographs of very small regions (4μ) showed that the hexagonal crystallites were oriented. The thickness of the carbon lamellae was estimated to be 13—23 Å. Two parallel exposures of an evaporated platinum layer indicated a resolving power of 20 Å for the electrostatic electron miscroscope.

(*Science Abstracts*)

Sommaire : La structure de la suie d'oxyde de graphite a été examinée au diffractographe électronique. Les éprouvettes furent métallisées par évaporation sous vide avec du platine ou de l'or.

Article by K. Beyersdorfer *Optik* 7, 1950 192-198

Machined Carbon Products 148/II

United Kingdom. Carbon can now be made available in a great variety of shapes and grades ranging from almost unmachinable tiles to very soft graphites. As a result the field of its application is constantly growing. One of its most useful properties is high resistance to chemical attack and this has recently been taken advantage of by making sealing rings from this material for mechanical seals, where contact is made with chemically active liquids. Another development of recent years is the use of carbon vanes in rotary pumps or compressors handling air or other gases which must not be contaminated. Such pumps are running dry; carbon is self-lubricating and makes it unnecessary to use oil as a lubricant. Pumps fitted with carbon vanes operate in a pressure range from 30 psi down to comparatively high vacua and have peripheral speeds reaching values up to 2,000 ft. per minute.

Sommaire : L'importance croissante du carbone en tant que matériau de construction pour l'appareillage industriel, par example des pompes rotatives est discutée.

Article by A. E. Williams *Mech. World* **130**, 3.8.1951 107-109

Electron Investigation of Glass 149/II

See Abstract No. : 144/II

Rare Metals in Electron Tubes 150/II

United Kingdom. The author surveys the metals used in valves and kindred apparatus discussing first structural materials then metals for plating and soldering and, finally, emissive materials and getters. Nickel is the most important of the structural materials, copper and molybdenum occupying second and third place. Tungsten is widely used for filaments and heaters, as leads sealed into glass and to a small extent for grid winding wires. Details of its production are given. Tantalum has been used in the past as a filament material but has now been replaced by tungsten. It is mainly used today as an electrode material. The principal plating metal is gold. In order to prevent electron emission in a valve from parts other than the cathode itself care has to be taken to keep the temperature of all affected components low. This calls for the application of highly conductive metals. It was found that plating of the grid wires with gold will largely prevent the formation of a film of cathode-coating materials on the grid. Gold is also used for making joints between copper surfaces and for final seals in valve assemblies. The most important emissive materials and getters are barium,

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caesium, thorium and zirconium. Reference is made to the production of these materials mentioning the Kroll process in the case of zirconium. Caesium has a work function lower than any other metal. Whilst this precludes its use as a thermionic cathode, it is applied in photo-electric surfaces of photo-cells, television camera tubes, etc. Details are given of the production of photo-electric surfaces. The article is well illustrated with diagrams, photographs and tables. One of the tables is reproduced below in abbreviated form.

Thermionic Properties of Metals

Metal	*Work Function ϕ (Electron volts)*	*Melting Point ° K.*	*Boiling Point ° K.*	*Temperature Te, where Vapour Pressure is 10^{-8}mm. ° K.*	$\phi / Te \times 10^3$
Aluminium	3.0	931	2,073	1,170	2.56
Antimony	4.0	903	1,690	730	5.5
Barium	2.4	981	1,420	680	3.52
Beryllium	3.9	1,623	1,723	1,100	3.54
Caesium	1.8	299	943	320	5.65
Chromium	4.6	1,890	2,470	1,040	4.42
Gold	4.9	1,336	2,773	1,200	4.07
Mercury	4.5	234	630	250	18.0
Magnesium	3.5	924	1,340	550	6.42
Molybdenum	4.4	2,833	3,830	2,230	1.97
Nickel	5.0	1,725	3,070	1,330	3.78
Palladium	4.9	1,822	2,570	?	—
Rhodium	4.6	2,213	2,775	?	—
Silicon	3.6	1,690	2,800	?	—
Silver	4.7	1,230	2,220	1,010	4.5
Tantalum	4.1	3,123	3,900	2,680	1.52
Titanium	3.9	2,070	3,275	1,600	2.44
Thorium	3.4	2,118	3,273	1,910	1.78
Tin	4.4	505	2,570	1,180	3.73
Tungsten	4.5	3,660	5,000	2,860	1.58
Zirconium	4.1	2,173	3,172	?	—

Sommaire : Compte rendu sur tous les métaux utilisés par la construction de tubes électroniques et appareils semblables.

Article by D. A. Wright *J. Brit. I.R.E.* **11**, Sept. 1951 381-392

Vacuum-Melted High Chromium Steels 151/II

See Abstract No. : 159/III

Gases in Cast Iron 152/II

See Abstract No. : 157/III

28 — APPARATUS COMPONENTS — 28

Glass Valves for Vacuum Control 153/II

United States. An all-glass valve is described for the admission of controlled amounts of gas to a vacuum system. It obviates the use of stopcocks – stopcock grease being a source of contamination – and can be operated at a pressure of 10^{-6}mm. Hg. The valve consists of two concentric tubes. One tube is joined to the vacuum system and the other is connected to the gas container. The material used is hard glass, of a type which matches the expansion co-efficient of the Kovar sleeve sealed to the outer jacket. The other end of the Kovar sleeve is sealed to the inner tube which carries a graded seal for connection to any pyrex glass vacuum system at the far end. A cut is provided in the inner tube which opens and closes as the Kovar seal is heated and cooled. An induction heater coil serves as the heat source. In operation, a heat of 200°C is applied which causes the cut in the inner tube to open and allows for the passage of the gas from the outer to the inner tube. When the desired amount of gas has been admitted the heat source is switched off and the contraction of the outer jacket causes the cut to close again.

Sommaire : Détails d'une vanne entièrement en verre pour l'admission de quantités dosées de gaz dans un système évacué.

Note by Anon. *Instruments* **24**, June 1951 656-659 see also *J. Franklin Inst.* **251**, June 1951 643-644

Vacuum Valve for the Handling of Very Pure Gases 154/II

United States. It is now possible to evacuate systems to a pressure as low as 10^{-9}mm. Hg. Where it is necessary to manipulate gases of extreme purity in such systems it is not practical to use greased stopcocks, as volatiles of the grease will tend to contaminate the system. An all-metal valve has been developed for this application. Its essential properties are : (a) It is air-tight down to pressures of 10^{-9}mm. Hg. Its leakage rate in the closed position is 10^{-11} litres per second. (b) It can be baked out at high temperatures. The valve con-

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sists essentially of two sections, (1) the valve assembly and (2) the driving mechanism. The seat of the valve is made from copper which fits into a Kovar cone. The Kovar cone travels over a total distance of 0.1″ at the rate of 0.01″ for each revolution of the handle. As a result a needle valve action is produced. In closing the valve a very large force can safely be exerted on the valve seat. The valve is assembled to the system by means of metal-to-glass seals. A valve as described has been used by the author for a period of several months, has been baked out more than 10 times and is still giving satisfactory service.

Sommaire : Une vanne mécanique entièrement métallique pour utilisation dans des circuits évacués à un vide extrême.

Letter by D. Alpert *Rev. Sci. Instrum.* **22**, July 1951 536-537

A Compact Sliding Vacuum Seal

155/II

Australia. The ordinary piping system of a vacuum plant consists of cylindrical piping elements which are joined by demountable seals. Where it is desirable to allow for translatory movement at the joints normal to the axis of the pipes, bellows have been used in the past or flanged joints which are lapped and greased. Metal bellows have the disadvantage that they allow for a small displacement only and lapped joints require frequent regreasing and relapping which is undesirable. The author describes an ' O '-ring type of joint allowing for translatory movement. The flange of one of the two pipe sections is fitted with a V-shaped groove to accommodate the ' O '-ring. The flange of the adjoining pipe section is recessed over a certain width of the flange. The depth of this recess is such that the two flanges touch each other, if the ' O '-ring is compressed by approximately 7%. The width of the recess is determined by the distance to be allowed for translatory movement. The actual displacement of one pipe unit against the other is carried out by push screws which are located in the flange provided with the recess. To allow for easy sliding of the ' O '-ring in the recess a small amount of a mixture of silicon grease with graphite is provided as a lubricant. The new joint has given satisfactory service and could be operated in systems kept at a pressure of 10^{-5}mm. Hg without breaking the vacuum.

Sommaire : Description d'un joint à anneau torique pour raccords tubulaires, lequel permet un mouvement de translation des deux tubes normalement à leur axe.

Note by R. I. Garrod *J. Sci. Instrum.* **28**, June 1951 187

Cold Trap Anti-Icing Shield

156/II

United States. Trouble has been experienced in the past due to the formation of ice from the atmosphere on the top of cold trap vacuum flasks attached to vacuum systems, especially where liquid nitrogen is used as a coolant. Details are given of a simple device which overcomes this difficulty. A shield fabricated from rolled sheet aluminium is placed over the top of the trap and is fixed at its upper end with a gasket made from rubber tape to the piping of the system. The diameter of the lower end of the shield is only slightly bigger than that of the diameter of the trap. As a result the evaporating coolant will escape at comparatively high speed through the air gap between shield and trap and will thus prevent the diffusion of atmospheric moisture into the flask. Moisture condensing on top of the shield does not freeze and runs off outside the vacuum flask. In the experience of the author a 20% reduction in coolant consumption is achieved, if a shield of this design is used.

Sommaire : Description d'un écran simple prévenant la formation de glace de l'atmosphère sur le haut des flacons à vide remplis d'axote liquide et formant pièges à congélation.

Note by J. E. Skvarla & E. C. Evans *Rev. Sci. Instrum.* **22**, May 1951, 341-342

29 — MISCELLANEOUS APPARATUS — 29

Storage Container for Liquid Helium

157/II

United States. Liquid helium is an excellent refrigerant for very low temperatures but it is difficult to prevent it from evaporating and it is equally difficult to transport it in the liquefied state. These difficulties have very largely been responsible for the limited use of helium in places, where no liquefaction plant is available. A storage bottle has now been developed which can keep liquid helium at —269°C for 100 days. The container described holds 10 litres, but larger sizes may become available in the future. The container consists of two concentric spheres made from copper, the inner sphere holding the liquid helium. The space between the two spheres has been evacuated. The double-walled sphere is immersed in a jacketted container which holds liquid nitrogen at a temperature of —196°C. The sphere containing the liquid helium is left uncorked to avoid explosions from the evaporating helium. The neck of the inner sphere holding the helium is kept very narrow, about ½-inch diameter. The new design is the outcome of an analysis of the heat flow into Dewar vessels previously constructed for the storage of liquid helium as described by Giauque. Largely due to the particular design of the neck, calculations predict an evaporation of the order of 0.1 litre per day. If the neck tube is coated with a layer of black paint to prevent the entrance of heat by radiation, this rate is reduced to 0.09 litre per day.

Sommaire : L'on donne des details d'un nouveau recipient de magasinage d'helium liquide, contenant 10 litres pendant 100 jours.

Note by A. Wexler & H. S. Jacket *Rev. Sci. Instrum.* **22**, April 1951 282 see also *Westinghouse Engr.* 11, July 1951 124

Preparation of Extremely Pure Helium Gas

158/II

See Abstract No.: 57/IV

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The Adsorption of Ultra-Violet Light as a Means of Detecting Leaks in High Vacuum Apparatus and Measuring Subatmospheric Pressure of Gases. 159/II

See Abstract No. : 137/II

Automatic Pressure Control Unit for High Vacuum Plant 160/II

Italy. A pressure control unit is described which consists of a Pirani gauge operating relays controlling the position of a valve situated in the line between fore pump and diffusion pump as well as the motor driving the fore pump. The unit has been incorporated in the pumping plant of a 1,100 kV. accelerator and covers the following operations : (a) It starts the rotary pump in the event, the pressure in the line connecting rotary and diffusion pump exceeds a given value. (b) It opens the valve connecting the two pumps as soon as the backing pressure required has been produced by the rotary pump. (c) It closes the valve between the two pumps and stops the rotary pump, when the pressure in the connecting pipe line has dropped below a given value and, finally, (d) it closes the valve, should the motor of the rotary pump fail.

Sommaire : Dispositif de contrôle de pression consistant en une jauge Pirani commandant la marche de la pompe à palettes et une vanne électromagnétique dans la tuyauterie vers la pompe à diffusion par le moyen de relais.

Article by M. Chiozzotto *Il Nuovo Cimento* 5, May 1951 345-348

Pressure Monitoring Device 161/II

See Abstract No. : 132/II

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30 — EVAPORATION — 30

Evaporation and Mobility of Naphthalene Molecules 134/III

See Abstract No.: 106/I

Evaporation in Vacuum by Means of Direct Electron Bombardment 135/III

France. A new method of heating the substance to be evaporated is proposed. It is an inherent feature of the conventional methods that the crucible is hotter than the processed material. As a result the crucible is liable to be attacked by the evaporating material. Here, the heating energy is applied directly to the substance. The temperature of the support or crucible is appreciably lower. The authors suggest bombarding the surface of the substance with electrons. The gun used for the purpose is of the type ordinarily incorporated in X-ray tubes. The processed material should be placed some 2 cm. away from the electron gun and should preferably be solid. Experiments with chromium in the form of sticks, and tungsten in the form of rods, have been successful.

Note by J. Brochard P. Giacomo P. Jacquinot & S. Roizen *J. Phys. Radium* **12**, May 1951 632

Sommaire: L'on propose de chauffer la substance qui est à évaporer par bombardement électronique pour éviter l'attaque du creuset ou du support par la matière évaporée.

Determination of Thickness of a Thin Film Obtained by Condensation of Artificial Radio-Active Elements 136/III

See Abstract No.: 108/I

Transparent Luminescent Films 137/III

United States. A brief description is given of two methods recently developed for the production of transparent luminescent films. The material best suited for the purpose is zinc sulphide activated with manganese. Details are given of the apparatus required. The films are deposited on pyrex plates by evaporation *in vacuo* and hydrogen sulphide is circulated through the chamber whilst the deposition is proceeding. The process is carried out at a pressure of a few mm. Hg and a temperature of 400 to 600°C. The thickness of the films can be controlled by observing the interference colours of the reflected light; the optimum value is 1 micron. This new development facilitating the production of luminescent films in transparent form opens up the way for a proper experimental study of electron penetration and scattering, and optical absorption.

Letter by F. J. Studer D. A. Cusano & H. Young *J. Opt. Soc. Amer.* **41**, Aug. 1951 559

Sommaire: Méthode de laboratoire pour la production de films luminescents transparents.

Variations of the Resistivity of Thin Metal Films as a Function of Thickness and Temperature 138/III

Switzerland. The electrical properties of thin silver films have been studied for various thicknesses and temperatures. Films with a thickness of less than 10 millimicrons show an irreversible increase of resistance, when heated up. Films of a thickness of approximately 12 millimicrons, however, show reversible changes of resistivity in strict proportion to the variation of temperature.

Article by J. P. Borel *Helv. Phys. Acta* **24**, 20.9.1951 389-400

Sommaire: Etude des propriétés électriques de couches minces d'argent.

The Light-Reflecting Power of Vapourised Aluminium-Silver and Aluminium-Magnesium Alloys 139/III

Germany. The reflecting power of films of metals, formed by condensation of vapours, was measured over the spectral range 2,500—5,000 Å. Data are given graphically for several concentrations of alloys. The reflecting power of pure aluminium is nearly constant over the range studied, at near 90% silver, and silver-rich alloys show very low reflection at shorter wavelengths. Pure silver shows a minimum under 10% at about 3,200 Å. For the longer wavelengths these alloys equal or exceed aluminium in reflecting power. At around 90% silver the reflecting power hardly exceeds 60% in the range studied, but 2% silver slightly improved the reflecting power of aluminium. Various concentrations of magnesium had only moderate effect on the reflecting power of aluminium. The curve for pure magnesium is similar to that for aluminium but a little lower.

(*Chemical Abstracts*)

Article by A. Boettcher *Z. Angew. Phys.* **2**, 1950 340-343

Sommaire: Les qualités réfléchissantes d'alliages aluminium-argent et aluminium-magnésium ont été étudiées.

The Application of Coatings to Optical Elements in Microscopy 140/III

Germany. The structure and the performance of reflection-reducing coatings applied to optical elements is discussed. Measurements of the actual increase of light transmitted through microscope lenses coated with an anti-reflection film have been taken by means of a photometer. The advantages offered by the application of such films to the optical components of instruments in particular microscopes designed for the study of fine details, are pointed out.

Article by H. Tronnier & H. Wagener *Optik* **8**, April 1951 165-172

Sommaire: Les avantages des film anti-réfléchissants pour le microscope optique sont soulignés.

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Checking the Quality of Aluminised Screens

141/III

See Abstract No. : 103/I

Aluminising Cathode-Ray Tubes

142/III

United Kingdom. The aluminium film deposited on the inside of the fluorescent screen in T.V. tubes fulfils five important functions : (a) light reflected backwards from the fluorescent screen is returned in a forward direction; (b) as the light reflected backwards is stopped from reaching the interior of the tube, no diffused reflection interferes with the contrast in the picture produced on the screen; (c) the occurrence of a ' high voltage rash ' is prevented; (d) the voltage of the screen is raised to that of the final anode which results in an improved brightness obtained on the screen; and finally, (e) no ion burns appear. The latter are noticed as dark patches in the centre of the screen which grow during the life of the tube and are caused by ions originated in the residual gas atmosphere of the tube. If the aluminium film is of the correct thickness, i.e. 0.1 micron, it will stop the passage of the ions, whereas it will allow the electrons to reach the screen. To assist the production of a film of the correct thickness an instrument has been developed which permits continuous thickness measurement of the aluminium deposit during the actual process of evaporation. It consists essentially of a variable radio-frequency oscillator, the inductive coil in the tuned circuit of which is mounted in a measuring head and placed close to the centre of the screen. The frequency indicating device is a fixed radio-frequency oscillator which is caused to beat with the output from the variable frequency oscillator by varying the capacitance in the tuned circuit accordingly. This oscillation is converted to the audio-frequency range and applied to a loud speaker or head-phones. The change in capacity required to balance the oscillation frequency in the measuring head resulting from the deposition of the aluminium film is a measure of the thickness of the film. The instrument can be pre-set to the critical value and thus work automatically.

Sommaire : L'on fait remarquer l'avantage d'aluminiser les tubes de télévision et il est décrit un instrument mesurant automatiquement l'épaisseur de la couche.

Note by S. R. Neuberger & J. H. Jupe *Wireless World* **41**, June 1951 245-246

Metallising Glass Components for T.V.

143/III

United States. Design features of a number of T.V. components are discussed which represent typical applications of conductor-to-glass processing. In the first instance, attenuator plates for microwaves are mentioned. The use of vacuum evaporation techniques for the production of the conducting film ensures high precision of dimensions and electrical stability. The resistance of the film is comparatively independent of frequency. A typical value is 140 ohms per square. Other components mentioned are capacitors made from ribbon glass and cylindrical components with a silver base, such as trimmers and inductances.

Sommaire : Un nombre de pièces détachées, utilisées dans les appareils de télévision, constitues d'éléments en verre métallisé.

Article by H. S. Craumer *Telev. Engng.* **2**, Sept. 1950, 16-17 & 23

Metallising Glass

144/III

See Abstract No. : 152/III

Metal Coating by Vapour Deposition

145/III

United States. A vacuum coating plant is described specially designed for the coating of strips of plastics such as cellophane or fabrics like paper, textile etc., all of which are readily liberating a large amount of volatile constituents during the actual deposition process. The evolution of gases is undesirable as it may interfere with the coating process and prevent uniform deposition. The plant consists essentially of three compartments through which the continuous band is passed. All compartments are properly sealed off from each other to prevent equalisation of pressures throughout the whole plant. The first compartment is heated in order to remove volatiles from the surface and body of the strip, but the temperature applied should not be so high as to cause embrittlement of the material. Passing into the second compartment the strip is cooled down by means of brine, liquefied gas or any other suitable refrigerant. The cooling of the band under a suitable sub-atmospheric pressure intensifies the removal of volatile components. Finally, the strip enters chamber No. 3, where the deposition takes place.

Sommaire : Les principaux éléments d'un appareil pour métallisation sous vide sont décrits. L'appareil comporte des dispositifs spéciaux permettant l'élimination des composants volatils d'un ruban sans fin en plastique ou textile à métalliser.

P. Godley & National Research Corp. *U.S. Patent* 2,562,182

Metal Finishing Plastics by Vacuum Coating

146/III

United Kingdom. Plastic components are replacing metal components on a steadily increasing scale, in particular, those of an intricate shape. Metallisation of such components – required for ornamental purposes in the case of jewellery, for instance, or for functional purposes in the case of electrical components – can be effected by four methods : (a) chemical silvering, (b) electroplating, (c) metal spraying, (d) vacuum coating. After a short discussion of the methods (a) (b) and (c), the author concentrates on an account of vacuum coating techniques and processes. The most popular coating metal is aluminium; its reflectivity is 89% as compared

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with that of silver=94% and chromium=65%. Due to the formation of natural oxide films the initial reflectivity value of aluminium is maintained far longer than that of the other two metals. The physics of evaporation are considered and details are given of the equipment designed for such processes. Evaporation is usually carried out at a pressure of 10^{-4} to 10^{-5}mm. Hg. Fast pumping is imperative to cope effectively with the volatile constituents of the plastics which may otherwise contaminate the pumping system. A speed of 1,500 litres/second is recommended. To reduce the amount of volatile constituents released from the plastic, the surface of the component is coated with a suitable sealing lacquer. This serves at the same time as a smooth base for the deposit ensuring a specular finish of the metal coating. Finally, in order to increase wear resistance a protective lacquer is applied after deposition. Here again the necessity for lacquering can be turned to advantage by adding various colour tints to the finished product. The author, then, proceeds to describe the deposition of films by cathodic sputtering. The application of this method is usually restricted to the deposition of precious metals and, whilst used on a large scale in the early days when it was difficult to produce low pressures, it is now confined essentially to the coating of plastic components which contain an abnormally high quantity of plasticiser. In conclusion, continuous coating processes are mentioned as applied in condenser production and, finally, the embossing of foils (cellophane).

Sommaire : Discussion de la métallisation des plastiques par évaporation et projection cathodique.

Article by L. Holland *Brit. Plastics* **24**, March 1951 96-102

Vacuum Metallising — 147/III

United States. The article is mainly concerned with the application of vacuum evaporation techniques in the coating field. Aluminium, gold, silver, zinc, chromium and nickel are the most popular materials used for evaporation. Vacuum coatings have properties which are useful for purely decorative as well as industrial purposes. Their high metallic lustre counts most in the former case and their thinness in the latter. Evaporated metal films are used for reflection, for partial reflection and partial transmission of light and heat waves, furthermore, for the conductance of electrical current and as barrier films to prevent the transmission of water vapour or other gases. Unlike electro-plating, metal coatings can be applied to non-conductors for instance, plastics and are sufficiently flexible to withstand bending of their support without losing adhesion or lustre. Among other applications, reference is made to gold stamping leaf. The process consists of depositing a wax layer, a layer of gold and finally a layer of thermo-plastic adhesive on a sheet of acetate or cellophane. The adhesive is brought into contact with the object to be stamped and a die brought down on the acetate sheet. The adhesive continues to adhere to the object and to the gold layer. The wax melts and separates the gold from the acetate sheet leaving a gold impression on the object. Using vacuum evaporation techniques for the deposition of the gold layer no more than 2.5×10^{-4} ozs./sq. in. are necessary for the production of a film less than three millionths of an inch thick. Finally, the author refers to masking techniques and metal coatings employed for the preparation of specimens. A brief discussion is added on the subject of capital expenditure for and the running cost of the necessary equipment.

Sommaire : Revue de la téchnique d'évaporation sous vide pour la métallisation de matériaux conducteurs et non-conducteurs.

Article by D. Rosenblatt *Mach. Design* **23**, Oct. 1951 141-144 & 180-181

Lustrous Metal Finishes — 148/III

United Kingdom. The merits of vacuum coating techniques in connection with the production of inexpensive jewellery are discussed. To quote one important advantage : vacuum coated plastic components need not be polished. Aluminium is used most frequently as the coating material. One pound of the metal is sufficient to cover 25,000 sq. ft. of work. A lacquer film is applied after coating to ensure good wear resistance (rather than protection from the effects of climate and atmosphere). This lacquer film can be used to give a variety of coloured finishes to the articles. If zinc is used as a coating material, its oxide covering the deposit will produce light interference patterns resembling mother-of-pearl. By depositing two metals of different colour on top of each other, the effect of an alloy can be achieved.

Sommaire : Description des techniques de revêtement métallique sous vide appliquées à la fabrication de fausse bijouterie.

Article by Anon. *Watchmkr. Jewllr. Silversmith* Aug. 1951, 70-73

Applications of Polystyrene Particles in Conjunction with Shadow-Casting to the Study of Polished Metallic Surfaces — 149/III

See Abstract No. : 145/II

The Method of Shadow-Casting in Photomicrography — 150/III

See Abstract No. : 119/I

Portrait Shadow-Casting — 151/III

See Abstract No. : 146/II

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31 — CATHODIC SPUTTERING — 31

Metallising Glass 152/III

Sweden. A survey is made of methods employed in the metallisation of glass for all applications, in household and industry. Burnt-in films are produced by roughening the glass surface and coating it with a metal paint which is subsequently burned in by heating the glass. Glass components treated in this manner are usually employed for decorative purposes. Metal spraying is ordinarily applied for the production of a relatively thick and highly resistant metal coating. The minimum thickness of a sprayed coating is about 0.02 mm. Deposition by cathodic sputtering is employed where relatively thin and bright metal coatings are required, e.g. in the case of optical mirrors, photo-cell barrier layers and high ohmic resistances. It takes ten minutes to deposit a silver film 0.0005 mm. thick. The technique is described in some detail. The table reproduced below is taken from the article :—

Vaporising Capacity of Metals in Hydrogen and Mercury Vapour

Hydrogen (850 V.) *Distance Cathode to Anode 6 cm.* *Cathode Area 7 cm²* *Current 40-50 milliamp*		*Mercury Vapour (100 V.)* *Distance Cathode to Anode 2 cm.* *Cathode Area 16 cm²* *Current 2-10 milliamp* *Temp. of Mercury 11-50° C.*	
Metal	*mg./amp. hr.*	*Metal*	*mg./amp. hr.*
		Be	0.43
Mg	9	Al	1.07
Ta	16	Mn	2.25
Cr	27	Fe	2.31
Al	29	Ni	2.40
Mn	38	Zr	3.39
Mo	56	Co	3.44
Co	56	Cr	3.49
W	57	Mo	3.92
Ni	65	Cu	6.89
Fe	68	Ta	7.40
Au	460	W	7.65
Ag	740	Pt	15.76
		Ag	20.47

It follows a short description of vacuum evaporation techniques employed in the manufacture of semi-transparent mirrors, optical mirrors, resistances, inductances on glass, etc. Vacuum evaporation techniques are indispensable where highly reflective aluminium films are required, e.g. in optical instruments. Very large objects have recently been coated by this method, e.g. the 5 m. diameter reflector of the Mount Palomar Observatory in U.S.A. Transparent protective coatings are also mentioned, i.e. thin silica films deposited by vacuum evaporation on front-surface mirrors. Finally, details of chemical and electrolytical procedures, are given.

Article by E. Umblia *Glass* **28**, Oct. 1951 343-351

Sommaire : Revue de tous les procédés de métallisation du verre, vaporisation, action chimique et techniques du vide.

36 — DRYING — 36

Drying of Pharmaceutical Powders and Crystals 153/III

United Kingdom. The overall efficiency and economy of 5 different drying techniques is discussed, i.e. oven driers, rotary driers, vacuum driers, drying by radiant heat, and high-frequency drying. For the purpose of comparison, it is assumed that these methods are applied to the processing of a specific heat-sensitive material in powder form with the object of reducing its moisture content from 0.5% to 0.01%. The following conclusions have been drawn. Oven drying is a widely established method but requires production in batches. Its power consumption is 14.4 kWh per ton. The main disadvantage of this method lies in the fact that a certain amount of heat is wasted as only the top of the material and to a lesser extent the bottom layer of the same is properly exposed to the heat. Rotary driers have only a very limited throughput, usually not exceeding 400 lbs. per hour. Vacuum driers are particularly suited for the treatment of heat-sensitive material. One can obtain higher outputs without increasing the temperature but procedures are necessarily confined to batch production. From the point of view of heating cost, this type of plant is economical, but the power cost is high and considerable maintenance work is required. Drying by radiant heat (usually in the infra-red range) allows for continuous production at a rate of 1,700 lbs. per hour. This type of plant is still in the development stage but although its power consumption is high its prospects are good. In the case of high-frequency drying, however, a number of practical difficulties have been encountered and have still to be overcome before this method will attract the industrialist.

Article by A. W. Kay *Mfg. Chem.* **22**, May 1951 188-189

Sommaire : Comparaison de 5 différentes méthodes de sèchage du point de vue d'efficacité globale et économie.

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Abstract No. and References

Chemical Engineering in the Frozen Food Industry

154/III

United States. The demand for frozen orange juice concentrate in the United States is steadily growing. The production in 1947-1948 amounted to 2½ million gallons=4% of the whole Florida crop. The estimate for the season in progress (1950-1951) is 28-30 million gallons=30% of the whole nation's crop. The progress of production methods in that field is typical for the progress in the frozen food industry as a whole and has been singled out by the author for a report reflecting developments in general. The production of frozen orange juice concentrate requires the application of high vacuum low-temperature methods on a large industrial scale. Major developments centre around the evaporator design and methods of freezing the product. Various evaporator constructions are discussed and shown in illustrations beginning with a falling-film recompression evaporator with multi-pass circulation, followed by an elaboration of this design which eliminates the necessity of recirculating within the stages. In addition, three evaporator constructions based on the heat pump principle are described and finally, a combination of 3-pass single effect recompression falling-film evaporators, consisting of three separate units, ' hooked ' together to operate as a single-effect evaporator. The processing temperature in all cases ranges from 55°F to 80°F. The second part of the article deals with developments of freezing methods, stressing the advantages of and latest advances in tunnel-type freezer construction.

Sommaire: Rapport sur le développement des méthodes de congélation et appareils employés dans la production de jus d'orange concentré gelé.

Article by V. C. Praschan *Chem. Engng. Progr.* 47, June 1951 325-330

37 — METALLURGICAL PROCESSES — 37

Vacuum Fusion Furnace

155/III

United States. A brief description is given of a furnace specially developed for gas determination by the vacuum fusion method. A pyrex glass envelope, 3-inches wide, contains a quartz tube of 2-inches diameter. The latter carries a graphite crucible supported on loosely packed graphite powder. A branch tube at a right angle connects with a series of small containers where the samples are stored prior to use. This arrangement facilitates the introduction of the sample into the actual furnace without breaking the vacuum. A magnet is used to transfer the sample from the storage container to the crucible. The pumping plant consists of a 2-stage mercury diffusion pump coupled with a mechanical fore pump. A run usually takes six hours. Operational temperatures are as high as 2,400°-2,650°C.

Sommaire: Description d'un four pour la détermination de gaz par méthode de fusion sous vide.

Note by Anon. *J. Sci. Industr. Res.* 10A, June 1951 259

Modified Vacuum Fusion Apparatus for the Determination of Oxygen, Hydrogen and Nitrogen in Certain Metals

156/III

United States. A vacuum fusion apparatus is described which has been specially designed to reduce the time required for each analysis. The new apparatus consists essentially of one melting chamber similar to that introduced by Guldner and Beach connected by means of a three-way stopcock to two identical measuring systems, each of which represents a complete unit consisting of a mercury pump, oxidising furnace, trap and calibrated volume. Ground glass joints are provided at all important points of the assembly. It is claimed that the new design doubles the capacity of the conventional vacuum fusion apparatus.

Sommaire: Un appareil de fusion sous vide est décrit, lequel consiste en deux systèmes de mesure doublant de la sorte la capacité des constructions habituelles.

Article by A. F. Torrisi & J. L. Kernahan *Analyt. Chem.* 23, June 1951 928-929

Gases in Cast Iron

157/III

United Kingdom. The occurrence of gases in iron and their effects on the properties of the same have been the subject of over 1,000 articles published in the last 50 years, but clarification of the problems connected with the presence of gases in metals is still lacking. For instance, there is no satisfactory method yet of examining metals for gas content in the molten state. As far as solid specimens are concerned the vacuum fusion method has proved highly satisfactory and the present article is essentially concerned with the determination of hydrogen, oxygen and nitrogen content in cast iron employing this method. The apparatus used in this study is similar to the apparatus described by H. A. Sloman and is coupled with a (modified) Ambler apparatus for the analysis of the gas samples. Discussing experimental procedure it is emphasised that the sample must be free from hydrocarbons, if accurate results are to be obtained. The specimen itself was prepared by cutting slugs from small bars cast in stick moulds of half-inch diameter and 5-inches length. In the author's opinion the accuracy of the method is within the following limits :—

Gas	*Average %*	*Degree of Accuracy %*
Oxygen	0.001 —0.025	±0.001
Hydrogen	0.0001—0.0020	±0.0001
Nitrogen	0.001 —0.010	±0.001

Two tables are given covering 31 different types of British pig-iron showing the gas content determined by vacuum fusion and, in addition, the content of major elements like carbon, silicon, etc., determined by chemical analysis. It can be seen that pig-iron with a high oxygen content contains at the same time a large volume of hydrogen. No definite conclusions are drawn from the position revealed in the tables. The examination of

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specimens taken from sand-cast and chill-cast bars gave identical results. The author finds that grey cast iron tends to retain hydrogen, possibly due to the presence of the graphite, whereas white cast iron releases hydrogen in appreciable quantities, when heated *in vacuo* to a temperature ranging from 600 to 800°C. Finally, the author calls for more research to achieve a better understanding of the part gas content plays in cast iron.

Sommaire : Discussion des techniques de fonte sous vide pour la détermination des gaz dans le fer et l'acier, particulièrement dans la fonte.

Article by R. V. Riley *Iron & Coal Trades Rev.* **162**, 11.5.1951 1083-1089

Production of High-Purity Iron and Iron Alloys on a 25lb. Scale

158/III

United Kingdom. An iron of very high purity was required for experiments in connection with a study of the effects of alloying elements on its mechanical properties. It was decided to use iron of a composition which could be purified by metallurgical methods as distinct from chemical procedures. The actual purification was carried out in two steps : firstly, melting in air, to reduce all oxidisable impurities, and secondly, melting in an hydrogen atmosphere, to remove the excess oxygen. A furnace holding a charge of 75 lbs. was employed in the first operation and a furnace holding a charge of 25 lbs. in the second operation. The latter was built into an air-tight tank and could be tilted inside that enclosure, together with the mould, by mechanical means controlled from outside. Iron melted in air, the product of the first operation, was placed in the furnace and the enclosure pumped down to a pressure of 0.001mm. Hg. Two rotary pumps were available working in series or parallel. After an interval of several hours, hydrogen was circulated through the enclosure at atmospheric pressure. Provisions were made to follow the progress of the reduction process. After completion of the latter, the hydrogen pressure was reduced to 5 cm. Hg and the melt poured into the mould. After melting in air the iron contained 0.28% oxygen and 0.01% nitrogen. Treatment in the hydrogen furnace reduced these figures to 0.001% or less and 0.001% respectively. Generally speaking the degree of purification achieved by this method was very satisfactory. Details are given in tabular form.

Sommaire : Description de méthodes et équipements employés pour la purification de fer consistant en une fusion à l'air, pour éliminer les impuretées oxydables, suivie d'une fusion en athmosphère d'hydrogène pour éliminer l'exces d'oxygène.

Article by B. E. Hopkins G. C. H. Jenkins & J. E. N. Stone *J. Iron & Steel Inst.* **168**, Aug. 1951 377-383

Vacuum-Melted High Chromium Steels

159/III

United States. Following up the pioneering work of Dr. W. Rohn, G. Hochmann melted chromium steels *in vacuo* and obtained a greatly improved toughness in steels containing 25% chromium. W. O. Binder and H. R. Spendelow examined the properties of vacuum-melted chromium steels and published a report which is the subject of this note. They found that the tensile strength of vacuum-melted steel was invariably lower than that of air-melted steel of the same chromium content but this could be compensated for by changing the percentage of chromium content. Impact values, however, differed greatly. In the case of 18% chromium steels impact values of 80-100 ft. lb. were established for vacuum-melted steels and 15-30 ft. lb. for air-melted steels. Embrittlement at room temperature in steels containing up to 40% chromium can be successfully eliminated but, if a higher chromium content were to be chosen, a reduction of the oxygen content below 0.04% would be necessary. The liquid metal has to be kept under vacuum until it has solidified as it reacts strongly with oxygen and nitrogen in the liquid state. In conclusion, it is stated that increased toughness and superior workability coupled with effective resistance to corrosion and scaling at high temperatures can be obtained by vacuum melting iron-chromium compositions, but there are still a number of difficulties to be overcome resulting from the low tensile strength and sigma-phase embrittlement.

Sommaire : Les propriétés d'acier au chrome de même composition mais fondu à l'air et fondus sous vide sont examinés et comparés. Le produit de la fusion sous vide présente une résistance à la traction inférieure, mais une résistance au choc et une ténacité nettement supérieure.

Note by Anon. *Engineer* **191**, 19.6.1951 864

Zirconium

160/III

United Kingdom. This is an extensive article on the production of zirconium, its properties and applications. The Kroll and Van Arkel processes are described. The mechanical properties of the metal are listed and details are given on its behaviour, when in contact with oxygen, nitrogen and hydrogen, etc. The table reproduced below is taken from this section. Several applications are mentioned : zirconium foil, 0.0005 to 0.002-inches thick, to be used as welding flux in connection with the welding of tungsten to tungsten or molybdenum, zirconium getters and zirconium secondary emission inhibitors, finally, surgical instruments made from zirconium and the zirconium vapour lamp.

Physical Properties of Zirconium

Atomic	1. Atomic number : 40 2. Atomic weight : 91.22 3. Lattice type : H.C.P. Below 880° C. a_0 =3.22Å c_0 =5.12Å c/a =1.589 B.C.C. Above 880° C. 3.61Å 4. Periodic classification group : IVA. Second long series

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Physical Properties of Zirconium—continued

	5.	Electronic structure : (2) (8) (18) (10) (2)
Mass		Density at 20° C. : 6.52 g./c.c.
Thermal	1.	Melting point : 1860° C. (3380° F.)
	2.	Linear coefficient of expansion : 20-200° C. ... ... 5.4×10^{-6}/° C. 20-400° C. ... ... 6.9×10^{-6} 20-700° C. ... ... 8.9×10^{-6}
	3.	Specific heat (0-100° C.) : 0.068 cal./gm.
	4.	Thermal conductivity at 50° C. (C.G.S. units) : 0.04 c.
	5.	Vapour pressure : No data (B.P. *ca.* 3000° C.)
Electrical	1.	Electrical resistivity : 39×10^{-6} ohm-cm. (iodide Zr)
	2.	Electrical conductivity (%) : 1ACS 4.1 (iodide Zr)
	3.	Temperature coefficient of resistivity : 0.004 C^{-1} (iodide Zr)
	4.	Superconducting at 0.7° K.
	5.	Electrochemical equivalent : 0.2363 mg./coul.
Thermionic	1.	Work function : 4.1 electron volts
	2.	Photoelectric threshold (long wave limit) : 3200 Å
Magnetic		Diamagnetic susceptibility :—0.45×10^{-6} at 18° C.

Article by G. L. Miller *Murex Rev.* 1, No. 8, 1951 184-194 see also *The Times* (*Industr. Suppl.*) Sept. 1951, 29

Sommaire : Exposé des propriétés, de la production et des emplois du zirconium.

38 — DISTILLATION — 38

Automatic Receiver Changer for Vacuum Distillation

161/III

Australia. A device is described which facilitates continuous product withdrawal from vacuum stills operating at very high reflux ratios. All fractions withdrawn are of the same volume. The apparatus has a cylindrical shape and consists of an upper and lower compartment. The upper compartment is fastened rigidly to the bottom of the frame work carrying the still and has a perspex cover. A tube leading through the cover conveys the distillate discharged from the still to a syphon tube which rests loosely in a bracket and can be of any desired size. The bottom compartment holds the test tubes awaiting filling. Each of the tubes is floating on a glycerol solution in a tubular container of its own. A motor is placed in the centre of the lower compartment which moves a funnel from one test tube to another. The funnel is bent and rotates on a thrust race round the vertical axis of the apparatus passing the distillate from the syphon tube to the test tube. An arm fixed to the funnel unit rests against the test tube whilst empty and acts as a location device. After the test tube has been filled it sinks to the bottom of the container and allows the arm to pass over it. The operation of the motor is controlled by a timing device. The apparatus has been successfully employed in connection with the operation of a 13 mm. Podbielniak hyperCal column operating at reduced pressure. It is necessary to allow for comparatively long ' off ' periods when timing the operation of the motor, to facilitate proper draining and to avoid loss of distillate.

Note by D. A. Simpson & M. D. Sutherland *Analyt. Chem.* 23, Sept. 1951 1345-1346

Sommaire : Description d'un appareil déstiné à être attaché à des appareils à distiller sous vide et qui recueille le distillat sans assistance.

Laboratory Apparatus for the Distillation of Oils for Diffusion Pumps

162/III

See Abstract No. : 127/II

Apparatus for Distillation on a Semi-Micro Scale

163/III

United Kingdom. Difficulties are often experienced in preparing or purifying small amounts of liquid, say, charges of a volume of 0.5 to 1.5 ml. Due to the liquid wetting the surfaces of the distillation apparatus, which are large compared with the quantity of liquid handled, an appreciable loss of material is incurred. A distillation apparatus is described which is designed to reduce this loss. The apparatus is recommended for charges of 1 ml. or less and can be used for distillation at atmospheric or at reduced pressures. It is made of pyrex glass and consists of a 'cold finger'-water condenser filled with cooling water. Provisions are made for the circulation of the same. The collector cup is hooked to two projections sealed to the bottom end of the condenser. This assembly fits into a distillation tube of about 25 mm. diameter and 150 mm. length. The latter is fitted with a thermometer pocket which is sufficiently long to prevent the vapours reaching the rubber sleeve which holds the thermometer. In the case of distillation *in vacuo* a different chamber is used fitted with one tubular outlet for the connection to the vacuum pump and another for the insertion to a leak-in needle valve. The thermometer pocket can be omitted, if the determination of the distillation temperature is not required.

Letter by J. M. Conolly & G. Oldham *The Analyst* 76, Jan. 1951 52-53

Sommaire : Détails d'un appareil à distiller permettant l'application de faibles charges sans perte appréciable en distillant soit à pression athmosphèrique, soit à pression réduite.

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Contd.

Abstract No. and References

A Rotary Thermal Rectifying Column

164/III

United States. The advantages and disadvantages of contact rectification and thermal rectification are discussed with particular reference to the pressure at which the distillation is carried out. The authors come to the conclusion that thermal rectification can be employed and yields sharp separation at reduced pressures, whereas contact rectification methods are not successful in the low pressure region. A new type of column operating in accordance with the principle of thermal rectification has been developed. It consists essentially of a cylindrical condenser made of metal, which is rotating, and a concentrically arranged glass column which is stationary. The condenser is cooled with water circulating through small copper tubes, fitted to the inside of the metal cylinder. The glass column is heated by means of a bifilar helical winding of heavy nichrome ribbon, which is applied directly to the outside of the tube and protected by a glass envelope. In operation, vapour rises in the annular space between the two tubes and the reflux stream returns on the inside of the outer tube in the form of a falling film. The heat applied to this tube causes partial vaporisation of the reflux and these vapours join the vapour rising in the centre. The condensate collecting on the metal tube is thrown off due to the rotary movement of the cylinder and is thus transferred to the reflux stream as a spray. A combination of these effects yields enrichment, if the vapour formed from the reflux is richer than the vapour stream at the same point or, if the condensate is poorer than the reflux it joins, or both. The performance of the new column is calculated for four pressure ranges (high, intermediate, low and very low). At high pressures the performance is much the same as that of ordinary columns, in the intermediate and low pressure range (approximately 0.01 mm. Hg) it is good but at very low pressures the performance is unsatisfactory.

Sommaire : Discussion des mérites respectifs de la rectification thermique en distillation à basse pression et description d'une colonne rotative en accord avec ce principe.

Article by E. S. Byron J. R. Bowman & J. Coull *Industr. Engng. Chem.* **43**, April 1951 1002-1010

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First Double-Flash Tar Distillation Units

165/III

Germany. Brief details are given of a new method for the distillation of tar. The new process is based on a method developed during the war which provided for distillation in two stages at atmospheric pressure. The first distillation process yields light oils, carbolic oils and naphthalene. The still is of the column type and is operated at a temperature of 330°C. The residue of this operation is transferred to a second column which is now vacuum operated and facilitates the distillation of wash oil and anthracene at temperatures as low as 320°C. The residue of the second distillation is an anthracene cake, the oil content of which can be reduced to 2% by centrifugation obviating the use of filter presses. The new process gives a 5% overall increase in oil yield over the previous method.

Sommaire : Description d'une nouvelle méthode de distillation de goudron utilisant des procédés de distillation sub-athmosphérique et atmosphérique.

Article by Anon. *Chem. Engng.* **58**, Aug. 1951 214-215 see also *Chem. Wk.* **69**, 14.7.1951 29

Equilibrium Phase Vaporisation of Petroleum Crude Oils or Fractions

166/III

United States. Vapour-liquid equilibrium relations for multi-component hydrocarbon mixtures are usually expressed by equilibrium flash vaporisation curves. The conventional method of determining these curves provides for the use of continuous vaporisers at atmospheric pressure but these units have several disadvantages. For instance, many petroleum fractions require handling at reduced pressures to avoid excessive temperatures resulting in decomposition. A new apparatus has been built which operates on the lines of a recirculating equilibrium still. The vapour rising from the boiling liquid is passed into a condenser, the condensate collected in a reservoir and finally recirculated into the boiling liquid. When conditions have stabilised, the vapour coming from the boiling liquid is of the same composition as the recirculating condensate. In this phase the temperature of the saturated vapour and that of the boiling liquid are constant and samples of liquid and condensate are taken simultaneously. The new still takes a charge of 500 ml. Details of design and operating procedure are given. To reach equilibrium conditions 0.5 to 4 hours are required depending on the nature of the material and the percent. vaporisation. Runs were made at vaporisation values ranging from 20 to 95%. Comparison of the performance of the new still with that of a continuous unit of conventional design proved that both types of units give results which are very close to each other. Eight different petroleum materials have been investigated by the new method. The pressures employed in these investigations varied from 3 to 760 mm. Hg and the temperatures from 100 to 675°F.

Sommaire : Présentation de la construction d'un appareil à distiller à recirculation équilibrée utilisé pour la détermination des points d'équilibre de phase d'évaporation de mélanges consistant en des composés multiples d'hydrocarbures devant être traités à basse température.

Article by D. F. Othmer E. H. TenEyck & S. Tolin *Industr. Engng. Chem.* **43**, July 1951 1607-1613

39 — MISCELLANEOUS PROCESSES — 39

Process of Producing Soluble Coffee

167/III

United States. A method is described for the production of a coffee powder which on re-constitution gives a beverage of controlled flavour and aroma. An important purpose of the process is to obtain useful by-products and to make them available for further application if not required for the production of the coffee powder itself.

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Abstract No. and References

The initial process consists of treating freshly pulverised and roasted coffee by solvent extraction using an aliphatic hydrocarbon as a solvent. In the second stage a continuously operated vacuum still is employed to facilitate the separation of the relatively volatile solvent from the fat and aroma constituents. The latter constituents are separated in a molecular still of the rotating disc type. The fat fraction can be used directly for edible purposes. The fat and aroma-free coffee may be extracted with a specific solvent for caffeine and subsequently reaches the final water extraction stage. The hot extract is concentrated by evaporation in vacuum and the resultant concentrate powderised in a spray drier. All constituents, separated in the process, in particular the aroma constituents, can be re-admixed to the actual coffee product in quantities as desired.

Sommaire : Description d'une méthode de production de café solubilisé basée sur l'emploi du vide à plusieurs stages.

R. Gilmont
U.S. Patent
2,563,233

Moveable Shelf Retort Dries Fish Quickly

168/III

United States. The fish to be fried is laid out on trays and placed on the shelves of a container which is lowered into a retort. The retort is then sealed and steam admitted at a pressure of 20 psi. Provisions are made for the steam to reach all parts of the chamber. After 30 minutes steam admission is stopped and the retort is exhausted to a pressure below atmosphere. At this stage the heat in the retort is still approximately 212°F. Consequently, the moisture contained in the fish is boiling off rapidly. The progress of drying is checked continuously by measuring the moisture content of the atmosphere in the chamber. When the moisture content has reached the desired low level, drying is discontinued and the vacuum is broken.

Sommaire : Description d'une méthode de sèchage de poisson, consistant en une cuisson sous 20 psi de pression, immédiatement suivie d'un sèchage sous vide grossier.

Note by
Anon.
Food Engng.
23, April 1951
190-191

Vacuum Refrigeration

169/III

Israel. It is known that a liquid boils at a temperature lower than normal if the pressure of the atmosphere surrounding it is reduced. Furthermore if the vapour formed during the boiling process is continuously withdrawn heat is removed from the liquid in quantities proportional to the amount of vapour withdrawn which results in a reduction of the temperature of the liquid itself. Vacuum refrigeration is based on these phenomena and has been known as a proposition for more than 20 years, but its introduction into practice was held up by a number of factors, in particular the absence of suitable pumps. The advent of steam-jet pumps made vacuum refrigeration an economic proposition for the production of drinking water or chilled water, for air conditioning, for the cooling of warm condenser water and cooling of aqueous solutions in the chemical and food processing industries. A vacuum refrigeration unit consists essentially of the following components : the vessel containing the water to be chilled, a steam-jet booster, a barometric condenser and a steam-jet pump. The latter maintains the vacuum required in the barometric condenser. The steam-jet booster evacuates the vessel containing the solution to be cooled by entrainment and transfers the vapours to the barometric condenser where the vapours are condensed by contact with cold water. The capacity of vacuum refrigeration plant is expressed in tons of refrigeration (1 ton = 12,000 B.Th.U. per hour). Units are available ranging in capacity from 20 tons to several hundreds. Ordinarily refrigeration units of this type work continuously but batch cooling is preferred where the cooling range is 100°F or more. The author was concerned for a while with studies on the recovery of potash from the Dead Sea. He points out that the use of salt brine available from the Dead Sea as cooling water for the condenser would reduce the capacity required for the steam booster appreciably as the vapour pressure of the salt brine is lower than that of water. At 50 feet below the surface of the Dead Sea the brine has a temperature of 20°C all the year round and therefore provides an inexhaustable supply of a cooling medium equivalent to water at 50°F. The use of barometric condensers avoids difficulties which may be encountered with other methods due to corrosion or scaling by the brine.

Sommaire : Les principes de réfrigération sous vide par jet de vapeur sont décrits, donnant en même temps des détails de la performance et des équipements existants dans le commerce.

Article by
D. Wittenberg
Int. Chem. Engng.
32, Aug. 1951
365-367
see also
Chem. Engng.
58, June 1951
141

IV — Special Subsidiary Subjects — IV

Abstract No. and References

40 — GASES AND VAPOURS — 40

Heat Transfer Through Gases at Low Pressures 53/IV

See Abstract No.: 113/I

Precision Multiple Sorption—Desorption Apparatus 54/IV

United States. The adsorption of gases or vapours on solids is usually determined by volumetric methods. However, gravimetric methods offer considerable advantages as they facilitate the simultaneous examination of several samples. The apparatus described here is a development of a design proposed by Stamm and Woodruff who used six silica spring balances which could be rotated in turn into position before a common measuring microscope. The new apparatus provides for fifteen silica spring balances mounted in individual glass tubes suspended in a thermostatically controlled bath. All readings are taken by a microscope which can be moved to successive balance positions by means of a hand-operated or motor-driven precision screw. The whole of the apparatus is made from glass; this includes the octoil oil diffusion pump. Other components of the apparatus are: an adsorbate source tube, a ballast volume, a differential manometer and a drying trap cooled with liquid nitrogen. In the majority of cases water vapour was employed as the adsorbate. The vacuum was measured with an ionisation gauge. At one time it was contemplated to use mercury cut-offs instead of grease-sealed stopcocks, but the performance of the apparatus was not markedly affected by a change to mercury cut-offs. Therefore, the use of stopcocks was continued. The apparatus described has given satisfactory service over a long period. Adsorption and desorption isobars and isotherms have been obtained for 500 samples in the temperature range from —20° to 60°C and at pressures from 10^{-6} to 400 mm. Hg involving 15,000 equilibrium point readings.

Sommaire: Appareil sorbant-désorbant permettant l'examen simultané de 15 échantillons d'adsorbants.

Article by W. O. Milligan, W. C. Simpson, G. L. Bushey, H. H. Rachford Jr. & A. L. Draper
Analyt. Chem. **23**, May 1951, 739-741

A Magnetically Compensated Vapour Density Balance 55/IV

See Abstract No.: 121/I

The Densities and Limiting Densities of Vapours 56/IV

See Abstract No.: 120/I

Preparation of Extremely Pure Helium Gas 57/IV

United States. Recent advances of cryogenic techniques have greatly assisted the development of processes concerned with the preparation of extremely pure gases. Extremely pure gases are required for the study of the interactions of electrons, ions and atoms in gases. This note reports on a new method of producing liquid helium with an impurity of the order of roughly one part in 10^{10}, whereas commercially available liquid helium has an impurity of one part in 10^4. Below the λ-point (2,186°K) helium has the character of a superfluid with zero viscosity. In the superfluid state it will penetrate minute openings which helium of a greater viscosity cannot penetrate. The new method of producing superfluid helium is based on the use of a ' superleak ' which consists of a platinum wire sealed into a pyrex capillary. An ' imperfect ' seal of this kind will permit penetration of superfluid helium only. The plant required consists of a vacuum system split into two sections. One section contains an ionisation gauge, which is employed for pressure measurement *and* for pumping to a level of 10^{-9}mm. Hg, pyrex (storage) bulbs and a small vessel incorporating the superleak mentioned. This section is connected to a diffusion pump coupled with a mechanical pump. A metal valve is provided to shut off the pumps when required. The second section, evacuated by a rotary pump, is formed by a Dewar vessel containing liquid helium, into which the superleak sealed to the first section is immersed. In operation the Dewar vessel containing the liquid helium is evacuated, until the temperature of the helium bath is reduced to about 2.15°K, well under the λ-point. The glass tip protecting the superleak is then broken and the superfluid helium diffuses through the leak from one section to another at a rate of roughly 0.2 cc/min. After about 3 cc. have been transmitted, the liquid helium in the Dewar vessel is warmed up to 4°K. This causes the superfluid helium transmitted into the other section to evaporate eventually filling the whole of that section at a pressure of nearly 760 mm. Hg. The pyrex (storage) bulbs are now filled with pure helium and can be sealed off.

Sommaire: Description d'une nouvelle méthode produisant du gaz hélium extrèmement pur.

Note by M. A. Biondi
Rev. Sci. Instrum **22**, July 1951, 535-536

Storage Container for Liquid Helium 58/IV

See Abstract No.: 157/II

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41 — FREEZE DRYING — 41

Freeze Drying 59/IV

See Abstract No. : 94/I

Sublimation of Ice in Vacuum 60/IV

Holland. In connection with an investigation into freeze drying generally, experiments were carried out on the sublimation of pure ice in vacuum. The experiments aimed at verifying the validity of a formula by Carman for the determination of sublimation rates (published in the *Trans. Faraday Soc.*, **44**, 1948, 529). A vacuum chamber was used incorporating a mechanical balance. At the end of one beam of the balance a thermally isolated copper tray was suspended in a vertical position. This copper tray carried the layer of ice and served as the evaporator. The change of weight resulting from the evaporation of the ice could be compensated by means of adjustment which were controlled from outside the chamber. The condenser was facing the evaporator at a distance which could be varied. The condenser surface was cooled with petroleum ether. The lowest temperature attainable was −70°C. The evaporator was heated by an electrical heating element which was placed at the back of the evaporator tray. The temperature of the evaporator and condenser could be measured with thermocouples and the amount evaporated by means of the aforementioned balance, the latter to an accuracy of 15 mg. Two series of experiments were carried out, one varying the distance between the evaporator and condenser from 12 to 44 mm. whilst the pressure was constant at a value below 10^{-3}mm. Hg, and the other varying the pressure between 10^{-3}mm. Hg and 0.4 mm. Hg. The results are given in tabular form. Good agreement was found between the sublimation rates calculated by the Carman formula and the sublimation rates determined experimentally.

Article by H. Kramers & S. Stemerding *Appl. Sci. Res.* **A3**, 1951 73-82

Sommaire : La formule de Carman pour la détermination des vitesses de sublimation a été confirmée par des essais sur la sublimation de la glace sous vide.

The Preservation and Clinical Use of Freeze-Dried Bone 61/IV

See Abstract No. : 125/I

Some Physical Factors that Influence the Survival of Brucella Abortus During Freeze Drying 62/IV

United States. Certain aspects of freeze drying technique as applied to the drying of mirco-organisms are still not fully explored. The authors investigated the influence of the temperature of the subliming ice, the rate of sublimation and the moisture content on the quality of the dried product. Brucella Abortus (BAI strain 19) was used as the test organism. The cells to be dried were suspended in a fluid of a composition similar to that recommended by Naylor and Smith (1946). Test results are given in terms of the percentage of the number of cells viable in the suspension originally and the number still viable after drying. The freeze drying apparatus employed in this experiment was of special design. It consisted of two chambers which were evacuated both by the same pump. The condensers were cooled with dry ice and the bottom of each chamber was fitted with a brass plate recessed to hold the bottles containing the test organisms. The temperature of the plate could be varied between −40°C and +50°C, by regulating the flow of cold or hot alcohol which was circulating through coils soldered to the bottom of the brass plate. If the temperature during processing did not rise above −30°C the percentage of viable cells recovered was high, but if the temperature was allowed to reach −10°C the rate of recovery was low. This seemed to indicate the existence of a critical value for the temperature. The presence of dissolved salts was observed to have an influence on the rate of destruction of cells which occurred, when drying was carried out at unfavourable temperatures. Further, there is obviously an optimum rate of drying. Generally speaking, the slower the rate of drying the higher the number of viable cells recovered. Finally, it was established that a correlation exists between residual moisture content and recovery of viable cells, i.e. at a high moisture content the percentage of cells, viable after drying and surviving a given period of storage, was low but this condition improved as dryness increased.

Article by R. S. Hutton R. J. Hilmoe & J. L. Roberts *J. Bacteriology* **61**, March 1951 309-319

Sommaire : Des expériences méthodiques ont été faites à fin de déterminer l'influence de facteurs tels que l'humidité du produit séché, la température de la glace en sublimation etc. sur la qualité de Brucella Abortus lyophilisées.

A Simple Apparatus for Drying Small Specimens of Sera 63/IV

Australia. A freeze drying apparatus of small capacity is described which can be built in the laboratory. It consists essentially of a manifold holding 52 ampoules, a condenser cooled with solid carbon dioxide, a vacuum chamber and pump, and a Pirani gauge. The vacuum chamber is built into a table of 60 × 55 cm. which is fitted with castors. The rotary pump is placed on a shelf below the table top. The manifold is clamped to a support and the whole assembly can be lowered into and removed from the vacuum chamber with ease. The rotary pump has a speed of 0.5 litres per sec. and produces a vacuum of 3 to 5 micron Hg. The manifold and condenser is made of pyrex glass tubing of 3 cm. diameter. The Pirani gauge has been ' fabricated ' from standard electrical parts. Each ampoule holds about 0.25 ml. of serum.

Note by J. F. Holden & J. Scott *Aust. J. Exper. Biol. Med. Sci.* **29**, 1951 47-50

Sommaire : Un petit lyophiliseur qui peut être construit par le personnel d'un laboratoire.

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

A Modified Freeze Drying Apparatus for Tissues

64/IV

United States. The application of the freeze drying technique to the preparation of tissues has the great advantage to eliminate exposure to solvents or protein precipitants. Details are given of an apparatus developed by the Medical Division of the University of Kansas Research Foundation. The frozen tissue is supported in a basket surrounded by an evacuated glass tube which is immersed in a Dewar flask containing 4 litres of liquid nitrogen to keep the tissue cool and provide a moisture trap. The pumping unit consists of a fore pump and an oil diffusion pump capable of producing a vacuum of 10^{-4}mm. Hg. The operating pressure is 10^{-3}mm. Hg. At this pressure the mean free path of water molecules is 6 cm. which exceeds the path the water molecule has to travel before reaching the moisture trap. The moisture trap, at the temperature of liquid nitrogen, leaves only 10^{-23}mg. of water per litre of gas at the above pressure as compared with 10^{-5}mg. in the case of drying with phosphorous pentoxide. Details of the mounting of tissue specimens are given.

Article by R. E. Stowell *Stain Technol.* **26**, April 1951 105-108

Sommaire : Discussion du séchage de tissus biologiques par lyophilisation.

45 — ELECTRONICS — 45

Rebuilding Boron-Wall Neutron Counters

65/IV

United States. Experience shows that boron-wall tubes usually fail for one of the following two reasons : the centre wire or end insulator has broken or a leak has developed allowing air to enter the tube. The author has repaired successfully a number of boron-wall neutron counters and at the same time has modified their construction. It is claimed that these alterations have extended the life of the counters considerably. The changes refer to the end cap design which is now made from rolled steel, a modification to the tension-spring suspension and the addition of an $\frac{1}{8}$ inch copper exhaust tube which is soldered to the end cap. When the tube is ready for pumping and filling, the exhaust tube is waxed to a vacuum system, the counter degassed and evacuated to 5×10^{-5}mm. Hg. The tube is then filled with a mixture of purified ethyl-ether (=0.7 cm. Hg) and neon (=8.8cm. Hg).

Note by E. H. Daggett *Nucleonics* **9**, Aug. 1951 72-73

Sommaire : Certaines modifications de la construction de compteurs de neutrons conventionels à paroi de bore sont discutées, lesquelles prolongeraient considérablement la vie du compteur.

On the Initial Decay of Oxide-Coated Cathodes

66/IV

Japan. Experiments are described which were conducted with the aim of clarifying the mechanism of initial decay of temperature-limited current from oxide-coated cathodes, over the 1/10th second time range. Commercial tubes have been used in the experiments having guard-ring anodes and electrolytic nickel cathode bases. It has been observed generally that the decay of the emission current became less pronounced after heating the cathode to high temperatures. Three experiments were carried out : (1) The bulb of the tube was heated from outside but the cathode was kept cold. As a result of heating the getter deposit on the glass walls, the decay was reduced to about one half of its initial value. However, the subsequent application of a high anode voltage caused the tube to revert to the previous state of decay. Repeated treatment of this kind was less and less effective. (2) The cathode was heated to high temperatures and the glass walls were kept cool. No change of the decay characteristics could be traced. (3) Special experimental tubes were used consisting of two compartments. One compartment was of similar size and construction as the ordinary measuring diode tube, the other served as a getter chamber not affected by the heat of the cathode. In this latter experiment high temperature heating of the cathode made no difference at all to the decay characteristics of the tube. The authors came to the conclusion that the initial decay of temperature-limited current from oxide-coated cathodes can be attributed to the poisoning action of gases present in the tube.

Letter by T. Hibi & K. Ishikawa *J. Appl. Phys.* **22**, July 1951 986-987

Sommaire : Le mécanisme de la chute initiale (1/10e sec) du courant limité par la température de cathodes revêtues d'oxyde est étudié et il est conclu que la chute de courant est due à des gaz polluents, présents. Le remède dépend de la capacité du getter dans le tube.

Rare Metals in Electron Tubes

67/IV

See Abstract No. : 150/II

47 — GLASS PROCESSING — 47

Glass-to-Metal Seal for Vacuum Systems

68/IV

United States. A simple means of sealing an ionisation gauge to the manifold of a vacuum system using slightly modified standard parts is described. A flare fitting and nut, and two ' O '-rings are required. The flare fitting, after some modification, is screwed into a tapped hole of the vacuum pipe and the joint sealed with glyptal or solder. Then, the two ' O '-rings and the nut are assembled to the glass tube of the gauge and the nut screwed into the flare fitting. A sketch is added showing details of the arrangement.

Note by F. B. Strother & H. Granberry *Rev. Sci. Instrum.* **22**, June 1951 432

Sommaire : Description d'un joint simple verre-métal fait avec des éléments standards.

IV— Special Subsidiary Subjects — IV

Contd.

Abstract No. and References

Glass-to-Metal Soldering

69/IV

United States. This is a short note describing a new process for the bonding of glass-to-metal which consists of painting the respective glass and metal areas with a thin layer of titanium hydride. Subsequently solder is applied and the assembly heated *in vacuo*. At a temperature of about 900°F the titanium hydride decomposes and causes the molten solder to adhere to the painted areas of the metal and the glass. A tight bond is formed upon cooling.

Sommaire : Description d'une technique nouvelle de soudure verre-métal.

Note by Anon. *Amer. Export. Industr.* 149, Aug. 1951 31

Vacuum-Tight Bonding of Mica to Glass

70/IV

France. A process is described of bonding mica foil as thin as 0.01 mm. to glass of ordinary composition. An enamel is used in powder form which softens at 360°C and melts at 550°C. The powder can be used also for the bonding of mica to metal alloys.

Sommaire : Description d'une technique nouvelle de jointure feuille de mica-alliages métalliques.

Article by J. Labeyrie & P. Léger *Le Vide* 6, Jan. 1951 951-952

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Amer. Export. Industr.	American Exporter Industrial, Johnston Export Publishing Co., 386, Fourth Avenue, New York 16, N.Y., U.S.A.
An. Mec. Elect.	Anales de Mecanica y Electricidad, Reina 31, Madrid, Spain.
Analyst	The Analyst, The Society of Public Analysts and Other Analytical Chemists, 7-8, Idol Lane, London, E.C.3, England.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 20th & Northampton Streets, Easton, Pa., U.S.A.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
ASEA Journal	ASEA Journal, Allmanna Svenska Elektriska Aktiebolaget, Vasteras, Sweden.
Aust. J. Appl. Phys.	Australian Journal of Applied Physics, Commonwealth Scientific and Industrial Research Organisation, 314, Albert Street, East Melbourne, C.2, Victoria, Australia.
Aust. J. Exper. Biol. Med. Sci.	Australian Journal of Experimental Biology and Medical Science, University of Adelaide, Adelaide, Australia.
Brit. J. Appl. Phys.	British Journal of Applied Physics, The Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
Brit. Plastics	British Plastics, Iliffe & Sons Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Canad. J. Res.	Canadian Journal of Research, National Research Council, Ottawa, Canada.
Canad. Min. Metall. Bull.	The Canadian Mining and Metallurgical Bulletin, Canadian Institute of Mining and Metallurgy, 906, Drummond Building, Montreal, Quebec, Canada.
Ceramic Industr.	Ceramic Industry, Industrial Publications Inc., 5, S. Wabash Avenue, Chicago 3, Ill., U.S.A.
Chem. Abstr.	Chemical Abstracts, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Chem. Age	Chemical Age, Benn Bros., Bouverie House, 154, Fleet Street, London, E.C.4, England.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. Wk.	Chemical Week, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Chemie Verlag, Weinheim/Bergstrasse, Hauptstrasse 129, Germany.
Chem. & Drugg. Export Rev.	Chemist and Druggist Export Review, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Chem. & Industr.	Chemistry and Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.J., U.S.A.
C.R. Acad., Sci., Paris	Comptes Rendus des Séances de L'Academie des Science, Messrs. Gauthier-Villar, Quai des Grands-Augustine 55, Paris, France.
Discovery	Discovery, Jarrold and Sons Ltd., London Street, Norwich, England.
Discuss. Faraday Soc.	Discussions of the Faraday Society, The Faraday Society, Aberdeen University Press Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Elect. Rev.	Electrical Review, Electrical Review Publications Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Electronic Engng.	Electronic Engineering, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Electronics	Electronics, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.

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Elektrotech. Z.	Elektrotechnische Zeitschrift, Verband Deutscher Elektrotechniker (VDE) Verlag, Wuppertal, Germany.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London, W.C.2, England.
Engng. Min. J.	Engineering and Mining Journal, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Food Manuf.	Food Manufacture, Leonard Hill, Ltd., 17, Stratford Place, London, W.1, England.
Foto-Kino-Technik	Foto-Kino-Technik, Foto-Kinotechnik G.m.b.h., Berlin-Borsigwalde, Eichborndamm 141-167, Germany.
Foundry	Foundry, Penton Publishing Co., Penton Bldg., Cleveland 13, Ohio, U.S.A.
Gen. Elect. Rev.	General Electrical Review, General Electric Co., 1, River Road, Schenectady 5, N.Y., U.S.A.
Glass	Glass, Glass Publications Ltd., 9, Moor Park Road, Northwood, Middx., England.
Glastech. Berichte	Glastechnische Berichte, Verlag der Deutschen Glastechnischen Gesellschaft, Untermainkai 12, Frankfurt (Main), Germany.
Helv. Phys. Acta	Helvetica Physica Acta, Physikalische Anstalt Universität Klinvebergstrasse 82, Basel, Switzerland.
Il Nuovo Cimento	Il Nuovo Cimento, Nicola Zanichelli S.A., via Irnerio 34, Bologna, Italy.
Indian J. Phys.	The Indian Journal of Physics, Indian Association for the Cultivation of Science, 2—3, Lady Willingdon Road, Jadavpur, Calcutta 32, India.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 33, Tothill Street, London, S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry, American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Instrumentation	Instrumentation, Minneapolis-Honeywell Regulator Co. (Industrial Division), Wayne and Windrim Avenue, Philadelphia 44, Pa., U.S.A.
Instruments	Instruments, Instruments Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Int. Chem. Engng.	International Chemical Engineering and Process Industries, Leonard Hill Ltd., 17, Stratford Place, London, W.1, England.
Iron & Coal Trades Rev.	Iron and Coal Trades Review, Industrial Newspapers Ltd., 49, Wellington Street, London, W.C.2, England.
J. Amer. Chem. Soc.	Journal of the American Chemical Society, American Chemical Society, 20th and Northampton Street, Easton, Pa., U.S.A.
J. Amer. Pharm. Ass.	Journal of the American Pharmaceutical Association, American Pharmaceutical Association, 2215, Constitution Avenue, N.W., Washington 7, D.C., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics. Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Bacteriology	Journal of Bacteriology, Medical Laboratories, State University of Iowa, Iowa City, Iowa, U.S.A.
J. Bone & Joint Surgery	Journal of Bone and Joint Surgery, 8, The Fenway, Boston 15, Massachusetts, U.S.A.
J. Brit. I.R.E.	Journal of the British Institution of Radio Engineers, 9, Bedford Square, London, W.C.1, England.
J. Chem. Educ.	Journal of Chemical Education, Div. of Chemical Education of the American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
J. Franklin Inst.	Journal of the Franklin Institute, The Franklin Institute of Pennsylvania, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Inst. Fuel	Journal of the Institute of Fuel, Institute of Fuel, 18, Devonshire Street, Portland Place, London, W.1, England.

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J. Iron & Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London, S.W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc., 29, West 39th Street, New York, 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J. Phys. Radium	Journal de Physique et le Radium, La Societe Francaise de Physique, 10, rue Vauquelin, Paris (5e), France.
J. Phys. Soc., Japan	Journal of the Physical Society, Japan, Faculty of Science, University of Tokyo, Japan.
J. Rech. Centr. Nat. Rech. Sci.	Journal des Recherches du Centre National de la Recherche Scientifique, 13, Quai Anatole-France, Paris (VIIe), France.
J. Sci. Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
J. Sci. Industr. Res.	Journal of Scientific and Industrial Research, The Council of Scientific and Industrial Research, India (Publications Div.), 20, Pusa Road, Delhi 5, India.
J. Sci. Instrum.	Journal of Scientific Instruments, The Institute of Physics, 47, Belgrave Square, London, S.W.1, England.
J. Soc. Chem. Industr.	Journal of the Society of Chemical Industry, Society of Chemical Industry, 56, Victoria Street, London, S.W.1, England.
Light Metals	Light Metals, Temple Press Ltd., Bowling Green Lane, London, E.C.1, England.
Mach. Design	Machine Design, The Penton Publishing Co., Penton Building, Cleveland 13, Ohio, U.S.A.
Mach. Lloyd	Machinery Lloyd, Continental Overseas Organisation, Ltd., 6, Cavendish Place, Regent Street, London, W.1, England.
Machinery	Machinery, Machinery Publishing Co., Clifton House, 83-117, Euston Road, London, N.W.1, England.
McGraw-Hill Digest	McGraw-Hill Digest, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Mech. World	Mechanical World, Kennedy Press Ltd., 31, King Street West, West Manchester 3, England.
Metal Prog.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metallurgia	Metallurgia, Kennedy Press Ltd., 31, King Street West, Manchester 3, England.
Mfg. Chem.	Manufacturing Chemist, Leonard Hill Ltd., 17, Stratford Place, London, W.1, England.
Microtecnic	Microtecnic, Scriptar S.A., 23, Avenue de la Gare, Lausanne, Switzerland.
Mod. Plastics	Modern Plastics, Modern Plastics Inc., 122, E. 42nd Street, New York 17, N.Y., U.S.A.
Murex Rev.	Murex Review, Murex Ltd., Rainham, Essex, England.
Mycologia	Mycologia, Mycological Society of America, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Nature (Lond.)	Nature, Macmillan and Co., St. Martin's Street, London, W.C.2, England.
Nucleonics	Nucleonics, McGraw-Hill Publishing Co. Inc., 330, W. 42nd Street, New York 18, N.Y., U.S.A.
Oil & Gas J.	Oil and Gas Journal, Petroleum Publishing Co., 211, South Cheyenne Avenue, Tulsa 1, Oklahoma, U.S.A.
Optica Acta	Optica Acta, International Commission of Optics, 3, Boulevard Pasteur, Paris (15e), France.
Optik	Optik, Wissenschaftliche Verlagsgesellschaft m.b.H., Stuttgart, Germany.

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Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London, W.C.2, England.
Phys. Rev.	Physical Review, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Proc. I.R.E.	Proceedings of the Institute of Radio Engineers, Institute of Radio Engineers, 1, East 79th Street, New York 21, N.Y., U.S.A.
Proc. Nat. Inst. Sci. India	Proceedings of the National Institute of Sciences, India, National Institute of Sciences of India, Delhi, India.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London, S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society. The Royal Society, Burlington House, Piccadilly, London, W.1, England.
Proc. Soc. Exper. Biol., N. Y.	Proceedings of the Society for Experimental Biology and Medicine, College of the City of New York, New York 31, N.Y., U.S.A.
PTT-Bedrijf	Het PTT-Bedrijf, Hoofdbestuur der Pasterijen, Telegrafie en Telefonie, Javastraat 6, The Hague, Holland.
Rech. Aéron.	La Recherche Aéronautique, Office Nationalé d'Etudes et des Recherches Aéronautiques, 3, Rue Léon Bonnat, Paris (16e), France.
Research	Research, Butterworths Scientific Publications, Ltd., 406, Bell Yard, Temple Bar, London, W.C.2, England.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	Science, American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Science Abstracts, Institution of Electrical Engineers, Savoy Place, London, W.C.2, England.
Sci. Rep. Res. Inst. Tohoku Univ.	Science Reports of the Research Institute of Tohoku University, Research Union Tohoku University, c/o Institute of Iron and Steel and other Metals, 75, Katahra Machi Sendai, Japan.
Slabopr. Obz.	Slaboproudy Obzor, Vocelova 3, Prague XII, Czechoslovakia.
Stain Technol.	Stain Technology, Biotech Publications, Geneva, N.Y., U.S.A.
Taylor Technol.	Taylor Technology, Taylor Instrument Co., Rochester (1) New York, U.S.A.
Telev. Engng.	Television Engineering, Bryan Davis Publishing Co. Inc., 52, Vanderbilt Avenue, New York 17, N.Y., U.S.A.
The Times (Industr. Suppl.)	'The Times' Industrial Supplement, 'The Times' Publishing Co. Ltd., Printing House Square, London, E.C.4, England.
The Times (Science Suppl.)	'The Times' Science Supplement, 'The Times' Publishing Co. Ltd., Printing House Square, London, E.C.4, England.
Trans. A.S.M.E.	Transactions of the A.S.M.E., The American Society of Mechanical Engineers, 29, West 39th Street, New York 18, N.Y., U.S.A.
Trans. Soc. Instrum. Technol.	Transactions of the Society of Instrument Technology, Society of Instrument Technology, 80, Bishopsgate, London, E.C.3, England.
Le Vide	Le Vide, Societe Francaise des Ingenieurs des Techniciens du Vide, 44, Rue de Rennes, Paris (VIe), France.
Watchmkr. Jewllr., Silversmith	Watchmaker, Jeweller and Silversmith, National Association of Goldsmiths, Heywood and Co. Ltd., Drury House, Russell Street, Drury Lane, London, W.C.2, England
Westinghouse Engr.	Westinghouse Engineer, Westinghouse Electric Corp., 306, Fourth Avenue, P.O. Box 1017, Pittsburgh 30, Pa., U.S.A.
Wireless World	Wireless World, Iliffe and Sons Ltd., Dorset House, Stamford Street, London, S.E.1, England.
Z. Angew. Phys.	Zeitschrift fur Angewandte Physik, c/o Springer Verlag/Berlin-Charlottenburg, Jebenstrasse 1, Germany.

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